

# STUDIES ON THE LOUSINESS IN SILK\*

By

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with 7 plates

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## I. Introduction

In the first report, the writer pointed out that the splitting of the silk fiber is a case of formation of the lousiness. In the second report, he also considered from the experimental point of view that the splitting is perhaps, influenced by these factors—the maturing degrees of silk substance in the silk-gland, the stretching multiple in spinning a thread out of the liquid silk, the differences in the spinning velocity, the relation between the weight of silk substance secreted in the gland and the weight of the silk spun, and the relation between the weight of the silk-gland and the size of an orifice of the spinneret. But no reference has yet been made to the manner in which such splitting takes place and to the mechanism by which it occurs. In order to get rid of the lousiness it is exceedingly necessary to investigate into the origination and mechanism of the lousiness and to find a way of prevention of it or the measures to remove it. This report includes the results of those investigations of both the origination of the lousiness in the course of treatments of silk fibers and their mechanism which have been carried out mostly by the writer himself.

In the writer's pursuit of these studies, many materials and advices have been given to him by Mr. Yamaguchi and Mr. Takeda, assistant professors of the Sericulture Course of the Textile Department of Shinshu University, to whom he would like to express his hearty thanks for their kindness.

## II. Experiments on the Splitting Quality of the Silk Fibers

### I. The Relations between the Degumming Degrees and the Splitting Quality of Silk Fibers and the Lousiness

The factors which have effects on the removal of sericin in the degumming process are the concentration of treatment solutions, the temperature and the time of treatment.

#### A. The Concentration of Treatment Solutions

Table 1. The Relation between Lousiness and Concentration of Soap Solution.

Concentration of soap solution	Layer of cocoon	10 min.	20 min.	40 min.	60 min.	80 min.	100 min.	120 min.
0.05%	Outer layer	0	0	0	0	0	0	0
	Middle layer	0	0	0	0	0	0	0
	Inner layer	0	0	0	0	0	0	0
0.1%	Outer layer	0	0	0	0	0	0	0
	Middle layer	0	0	0	0	0	0.2	0.6
	Inner layer	0	0	0	0	0	0	0.2
0.5%	Outer layer	0	0.4	0	0	0.2		0.2
	Middle layer	1.0	1.2	2.8	1.8	3.0		3.0
	Inner layer	0	0.8	0.4	0.2	0.8		0.2
1.0%	Outer layer	0	0	0.6	0.4	0.4		0.6
	Middle layer	1.4	2.6	7.6	4.0	3.4		6.2
	Inner layer	0	0.2	0.4	0.4	1.8		1.8
2.0%	Outer layer				0			0
	Middle layer				2.0			0
	Inner layer				0			0
4.0%	Outer layer				0			0
	Middle layer				2.0			3.0
	Inner layer				0			0

Remarks: Number of lousiness is the one in 10 cm of cocoon fiber, tests are done with 100 times microscope.

The lousiness appears variously according to the different grades of concentration of the treatment solution; the lousiness most shows itself in the concentration of from 0.1 to 1.0% used usually in the degumming.

In the case of the lower grades of concentration, the degumming action is weak and the splitting of the fiber rarely takes place and consequently there are very few spots of lousiness produced. On the other hand, the higher grades of concentration of the solution used in the treatment lessen the lousiness; the reason of this fact is that the fibrils once generated are broken longitudinally by the strong action of the solution and then are dispersed into small pieces in the solution. Therefore, it can be said that the

reagent of the higher grades of concentration give impetus to the longitudinal breaking of the silk fiber.

### B. Treatment Temperatures and the Origination of the Lousiness

The cocoons of the silkworm kinds, European No. 19×Chinese No. 17, were used; the three kinds of the cocoon layers, i.e. the outer, middle, and inner layers were reeled off respectively. Each skein of 50-threads thus reeled was wound on a glass frame, 10 cm in width and then treated with distilled water for three hours at the temperatures shown in the following table. As a control for comparison, the soap solution of 0.5% concentration, fifty times more than the weight of the sample, was used at the temperature of 97 to 98 C. for 80 minutes of treatment. The observation was made with a 100-times microscope, after the threads had been removed from the glass frame. Each of the fifty degummed threads of length of 10 cm. was examined for lousiness and split fibers.

In order to make the observation easy, the samples were colored with Soler-cyanin (acid dye) and Rhodamin B conc (basic dye).

Table 2. Relation between Lousiness and Temperature in Treatment.

Classification	Outer layer			Middle layer			Inner layer		
	Boil.-off loss	No. of lousiness	Separate fiber	Boil.-off loss	No. of lousiness	Separate fiber	Boil.-off loss	No. of lousiness	Separate fiber
Control	26.6%	15	##	16.3%	56	##	14.6%	0	+
100°C	25.3	29	##	18.5	61	###	15.1	0	+
110 "	27.4	32	###	18.7	63	##	15.9	2	##
120 "	29.4	49	##	20.5	71	###	16.3	0	+
130 "	31.8	20	##	22.5	21	##	20.0	2	+

### C. Treatment Time and the Origination of the Lousiness

Only the outer layer of silk kind of the European No. 19×Chinese No. 17, was taken as a sample, and treated in distilled water at such temperatures and for such time of treatment, as in the Table 3.

Table 3. Relation between Lousiness and Time of Treatment.

Temp. Items Time	80°C			100°C			120°C			130°C		
	Boil-off loss	No. of lousiness	Separate fiber	Boil-off loss	No. of lousiness	Separate fiber	Boil-off loss	No. of lousiness	Separate fiber	Boil-off loss	No. of lousiness	Separate fiber
Control	— %	—	—	26.6 %	15	##	— %	—	—	— %	—	—
1 hr.	—	—	—	—	—	—	—	—	—	—	85	###
3 hrs.	—	—	—	—	—	—	—	71	###	—	50	##
5 "	—	—	—	—	—	—	—	53	##	—	15	+
10 "	15.3	0	+	24.8	36	##	29.6	38	##	46.2	0	+
20 "	20.5	1	+	27.1	64	###	41.6	17	++	50.4	0	—
30 "	24.4	18	++	31.5	31	##	46.4	6	+	60.9	0	—
50 "	22.8	40	##	34.6	24	++	63.5	0	+	69.3	0	—
60 "	24.5	52	##	—	14	—	69.5	0	—	—	—	—
70 "	—	30	##	—	7	—	—	0	—	—	—	—

The Relation between lousiness and time of treatment for each temperature is shown in Fig. 1.

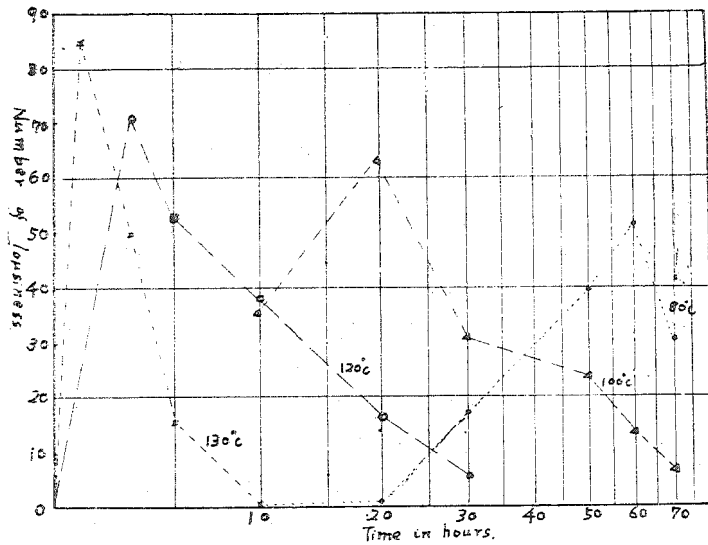


Fig 1.

The above experiments have shown that the silk fibers differ in their splitting quality according to their parts, and also that there are some kinds of silkworms the fibers of which are hard to split and there are also other kinds of silkworms the fibers of which are easy to split. In other words the former kinds have the tendency to produce very slight lousiness in spite of any treatments and to make very few changes under treatment conditions, while the latter kinds increase the splitting and the number of the spots of the lousines in any progress of the treatments until the maximum point is reached, after which the lousiness gradually decreases and finally disappears.

The cause of this disappearance is that fibrils once produced by splitting are further broken lengthwise, falling off the masses of fibers. In the course of these treatments, the silk fibers become gradually deteriorated.

This is probably because non-crystalline parts of the silk fibers dissolve and fall off in the solution. Any changes both in the temperatures and in the time of treatment produce the differences of the appearances of the lousiness; in other words, the higher the temperature, the shorter the time in which the lousiness shows itself most.

The maximum points observed in the experiments are as follows.

Temp.	Treatment Time	No. of lousiness
80°C	60 hours	52
100	20	64
120	3	71
130	1	85

If truly the lousiness or separate fibers consist of the so-called branched fibers laying in the cocoon fibers as many investigators reported, the same results of the appearance of the lousiness should be got in this experiment whatever the treatment may be. Accordingly it must necessarily be thought that the variability in the appearance of the lousiness is influenced causatively by the differences of the splitting qualities of the silk fibers. The above experiments also show that the speediness or slowness of the appearance of the lousiness is caused by the treatment conditions.

Within the limits of the practical degumming conditions, the number of the spots of the lousiness indicates an increasing trend in accordance with the time of treatment. It varies also by dint of the changes of the concentration of the treatment solution. In order to know whether the origination of the lousiness in the different temperatures and time above mentioned takes place also in each of the different layers of a cocoon, the observation was made of the outer, middle, and inner layers respectively, as shown in the next table.

Table 4.

Time	Cocoon layer Items	Temp.		80°C		100°C		120°C		130°C	
		Boil-off loss	No. of lousiness	Boil-off loss	No. of lousiness	Boil-off loss	No. of lousiness	Boil-off loss	No. of lousiness		
10 hrs.	Outer 1.*	15.3%	0	24.8%	52	29.7%	35	46.2%	1		
	Middle 1.	11.1	2	17.3	10	23.3	28	34.6	33		
	Inner 1.	11.3	1	18.0	1	25.1	1	33.8	1		
20 hrs.	Outer 1.	22.0	20	27.2	112	41.6	23	50.4	10		
	Middle 1.	13.5	2	20.7	40	32.8	1	47.5	1		
	Inner 1.	13.8	1	19.0	3	33.9	0	42.9	1		
30 hrs.	Outer 1.	24.4	58	31.5	60	46.4	9	60.9	0		
	Middle 1.	15.4	5	25.4	27	45.5	0	59.6	0		
	Inner 1.	14.9	0	22.6	0	44.5	0	50.5	0		
50 hrs.	Outer 1.	22.8	60	34.6	70	63.5	0	69.3	0		
	Middle 1.	16.5	19	28.4	0	65.5	0	62.3	0		
	Inner 1.	16.1	0	26.3	0	51.7	0	57.9	0		

Remarks: Kinds of cocoons and methods of degumming are the same with Table 3.

\* 1. ...Layer.

From the results shown in Table 4, it is to be noted that, according to the manner of partitioning the cocoon layers, the lousiness appears most in the outer layer or in the middle layer. Just like the results of the experiments hitherto carried out, the results shown in Table 4 have shown that the part of the cocoon layer hard to split has low variability, but the easily splitting part has high one.

From the results of the above experiments, it can be affirmed that the principal cause of lousiness is the splitting quality of the silk fibers.

## 2. The Splitting Quality of the Main Fibers

How far the silk fibers are split and disintegrated is unknown, but it is assumed that they go electron-microscopically,<sup>(3)</sup> so far as to their micellar structures. The following experiments were carried out to know how far the splitting takes place by the use of the ordinary treatment. A skein of 50-threads, reeled from cocoons of the lousiness-possessing kinds, European No. 19 × Chinese No. 17 and subjected to the regular degumming, was taken

as a sample. The examinations were made of the fibrils separated from the main fibers by steeping the sample in a lousiness-removing reagent, the composition of which is shown below. The various split fibers produced in the course of the second degumming after the removal of the lousiness appeared during the first degumming and the number of the spots of lousiness were also examined.

The composition of the lousiness-removing reagent is

CuSO <sub>4</sub>	NaOH	Glycerin	H <sub>2</sub> O
16 g	15 g	8 cc (S.G. 1.265)	100 cc

The copper glycerin solution of such proportion as above was prepared, and 12 cc of glycerin was added to 30 cc of the original solution in order to adapt to the proper treatment for the lousiness. In using it one part of this solution was further diluted to 30 parts with distilled water. This is called "G-solution" here. The samples were treated with this solution at 50°C. for each time. After that, each sample was given an intense re-degumming process with the soap solution of 0.5% conc., the temperature of 98°C. and the time of 6 hours. There lousiness was examined with a 60-times microscope.

Table 5.

Time of treatment by G-sol <sup>a</sup>	No. of times	Original sample		After treatment of G-solution		After re-degumming	
		No. of lousiness	Separate fiber	No. of lousiness	Separate fiber	No. of lousiness	Separate fiber
2 min.	1	18	###	0	###	0	###
	2	17	###	1(small)	###	0	###
5 min.	1	14	###	0	##	0	##
	2	16	###	0	+	0	+
8 min.	1	22	###	0	+	0	+
	2	12	###	0	+	0	+
10 min.	1	17	###	0	+	0	+
	2	12	###	0	+	0	+
15 min.	1	18	###	0	+	0	—
	2	6	###	0	—	0	—
20 min.	1	9	###	0	—	0	+
	2	13	###	0	—	0	—



In this experiment, when the lousiness which appears in the original sample is treated with the G-solution, it resolves and vanishes. Separate fibrils produced in a short-time treatment still remain in the solution, but a longer treatment makes them vanish away in it. This is due to the differences between the thickness of the separate fine fibers of the lousiness-mass and the thickness of the other separate fibers. For the fibers the lousiness of which vanishes are always finer than the ones the separate fibrils of which remain.

It was noticed also that the samples after the re-degumming process showed almost the same appearance of separate fibers in the case of a short-time treatment with the G-solution as the previous samples, but the gradual decrease of the separate fibrils goes with an increase in the treatment time. The progress in the G-solution treatment makes separate fibers of rather thick size disperse and vanish away in the solution, and it is thought that the further degumming of more intensive degrees does not cause separation of the fibers, but the fibers which have already separated begin at once their breaking lengthwise.

The following table shows the results of the degumming by the use of an autoclave.

Table 6.

Degumming condition	No. of measurement	Original sample		After treatment of G-solution		After re-degumming	
		No. of lousiness	Separate fiber	No. of lousiness	Separate fiber	No. of lousiness	Separate fiber
10 hrs/100°C	1	28	###	0	+	0	+
	2	12	###	0	+	0	+
5 hrs/110°C	1	31	###	0	-	0	##
	2	20	###	0	-	0	##
3 hrs/120°C	1	15	##	0	+	0	##
	2	18	##	0	+	0	##
2 hrs/130°C	1	16	##	0	+	1 (small)	##
	2	16	##	0	+	0	###

Remarks: Time of treatment by G-solution is 5 minutes, Temperature 50°C.

The treatment conditions for the temperatures in this table are the most suitable for the appearance of the lousiness shown in Table 3.

In this case also the lousiness masses fall off completely by the G-solution steeping, but the differences are due to the fact that the rising of temperature seems to cause the severe splitting of the silk fibers. This fact shows that the rising of temperature in Table 3 causes an increase of the absolute value of the lousiness i.e. the degumming temperature is connected with the splitting quality. It was thought that the fact the changes of the conditions of degumming bring forth the differences of the splitting means that the more intense degumming increases split fibers in number and changes the aspect of the lousiness. Then the experiments were made under the following conditions:

**The method of degumming:**

Temperature used, 120°C.; the pH of water (cold) used was 6.8, and became 8.4 when heated to 100°C.; period of time, 1-5 hours.

**The samples used:**

Sample A; this sample consists of those threads abundant in lousiness which were subjected to the regular degumming and examined about their lousiness and then given secondary the above-mentioned degumming.

Sample B; this sample was subjected to the same degumming as sample A and treated with G-solution (Temp. 50°C., Time 5 minutes) and then given secondary the degumming under the above conditions.

Table 7.

Time of treatment Samples	Items	Original sample		After treatment of G solution		After re-degumming		Remarks:  (After re-degumming)
		No. of lousiness	Separate fiber	No. of lousiness	Separate fiber	No. of lousiness	Separate fiber	
Sample A	1 hr.	34	##	—	—	16	###	Lousiness of small shape become dissolved. I have observed the split of fibroin in each part.
	2 hrs.	42	##	—	—	24	###	id. split forms of lousiness are observed.
	3 hrs.	45	##	—	—	28	###	id. small shapes of lousiness are observed.
	4 hrs.	36	###	—	—	22	###	Lousiness of small shape become dissolved, lousiness of split form become numerous.
	5 hrs.	43	###	—	—	25	###	id.

Sample B	1 hr.	43	≡	0	+	5	≡	Only split form of lousiness are observed I have observed lousiness of split form of main fibroin.
	2 hrs.	46	≡	0	+	—	—	No redegumming.
	3 hrs.	41	≡	0	+	4	≡	Split form of lousiness is numerous, split fiber arises in short length of fiber.
	4 hrs.	45	≡	0	+	—	—	No re-degumming.
	5 hrs.	20	≡	0	+	5	≡	The same with column of 3 hrs.

As the writer anticipated, there was an increase of split fibers and also of the lousiness. From this result, it is assumed that in Sample A the number of the lousiness decreased, because the small spots of lousiness disperse and vanish in the solution by the re-degumming and there remain only large ones, almost all of which are the split types (bundled or split types stated in the first report). And the split fibers do not distribute themselves uniformly on the sample; their appearance is different according to the position. This shows the partial differences of the splitting quality of the fibers. This is remarkable in Sample B, i.e. the lousiness and separate fibers which at first emerged vanish for the most part on account of the G-solution treatment, and again emerge after the re-degumming; and both the lousiness and split fibers increase in number, the shape of the lousiness belonging almost to the split types. By further increase of the treatment time all the lousiness and split fibers disperse and vanish. (table omitted)

From the above fact, it has been assumed that in the silk fibers the splitting in the main fibers takes place during the degumming process. All of the silk fibers, however, have not the same splitting quality.

The lousiness has a close relation with the structure of the cocoon filaments. There is essentially high or low splitting quality in silk fibers. In the first report, the writer divided the lousiness into two types according to its shape i.e. a split type (append. Fig. 1) and a bundled type (append. Fig. 2); the former is of large size and the latter is of small one. The procedure of splitting of the main fibers is illustrated in the appendant Fig. 3 and Fig. 4.

### 3. The Maturing Degree of the Silkworm and the Splitting Quality of Silk Fibers

In the second report, it was reported by the writer that the lousiness increases in number in accordance with the growth of the silkworm, which the writer divided into an under-grown, a grown, and an over-grown state. And it was described that this increase is due to the differences in the

maturing degrees of liquid silk when it is spun by the silkworm. In this section, the emergence of the lousiness was examined with materials devised in various ways. Seihaku, and Japanese No. 115×Chinese No. 108, and Sanmin-san which spins a cocoon after the third moulting were taken as materials and were cocooned in the following way:

a. **Spiracle-closing Silkworms**

These silkworms secreted the same amount of silk substance as the control-silkworms but their cocoon-fibers spun from the spinnerets are thicker.

b. **Over-grown Silkworms**

The silkworms started to build cocoons but then they were stopped spinning for 24 hours or 48 hours and they were again cocooned after that stoppage. Their cocooning was stopped by means of the writer's method. Measurement was made in the following way:

Remarks:

L.S. :—Lousiness-spots. Real measurements of the lousiness-spots appeared on each of 50 cocoon-fibers which are 10 cm in length.

S.F. :—Split fibers

As it was difficult to observe them in their proper state of split fibers, they observed as separate fibers.

B.F. :—Branched fibers

Real measurements of 50 cocoon-fibers in a cocoon-layer.

Table 8.

Kind of cocoon	Layer of cocoon Items Sample	1st layer			2nd layer			3rd layer			4th layer			5th layer		
		L.S.	S.F.	B.F.	L.S.	S.F.	B.F.	L.S.	S.F.	B.F.	L.S.	S.F.	B.F.	L.S.	S.F.	B.F.
		Seihaku	Control	0	—	0	0	+	0	0	≠	0	0	—	0	0
Closing 1st spiracle	0		—	0	0	+	0	0	+	0	0	+	0	0	+	0
Over-Maturity I	0		+	0	1	≠	8.7	0	≠	1.5	0	≠	1.0	—	—	—
Over-Maturity II	6		≠	9.9	2	≠	2.6	1	≠	3.0	—	—	—	—	—	—
Sanmin-san	Control	0	—	0	0	—	0	2	≠	0	7.0	≠	7.5	0	≠	0
	Closing 1st spiracle	0	+	2.0	0	≠	0	6	≠	0	1	≠	0	2	≠	0
	Over-Maturity I	0	—	15.5	16	≠	10.2	8	≠	3.7	2	≠	3.5	—	—	—
	Over-Maturity II	0	≠	13.5	17	≠	10.5	2	≠	4.5	4	≠	3.5	0	+	0

Remarks: L.S.....Lousiness-spot. S.F.....Separate fiber.  
B.F.....Branched fiber.

As seen in Table 8, in the control of the race Seihaku, no lousiness emerges owing to the degumming, but if it is cocooned when over-grown, the splitting quality of its fibers increases and also the lousiness increases. From this result, it can be said that the splitting quality is determined when the fibers are formed. Therefore, the origination of the lousiness is thought to be structural.

That the cocoon-fibers of the spiracle-closed silkworm are low in splitting quality and are scarce in lousiness, is thought to be due to the weak oppression of the silk-press and to the lowering of the fiber-forming ability, considering that these fibers are thicker in size than the control. On the other hand, it is assumed that when cocooning time is prolonged, spinning velocity becomes slow. As seen in the above description, there are two ways of increasing lousiness: promotion of the maturing degree of the silkworm and that of the degumming degree (Table 3). The results shown in Table 3 are different from those of the three kinds mentioned in Table 8 i.e. in the degumming test no change of the number of the lousiness can be seen both in the case of the race Seihaku and in the case of the control and the test-division of the so-called three-moulting silkworm, while in the case of the kind Japanese No. 115×Chinese No. 108 there appears much lousiness in the test-division. On the other hand, by the method of promoting the maturing degree, each of the three kinds has much appearance of lousiness. This fact accounts that the splitting quality of the fibers are dominated by the condition of silk formation. And it is assumed that the better orientation of micells in the silk fibers makes the higher splitting quality. It is also said that better micell orientation in the silk fibers is obtained by the over-grown silkworm.

#### 4. The Shapes of the Spinnerets and the Splitting Quality of Silk Fibers

In the second report, the writer reported that the size of the spinneret has some relation with the origination of the lousiness. He also perceived that, from his studies in the silkworm's spinning mechanism, there are some differences in the spinnerets, especially in the constructions of the silk-presses according to the kinds of the silkworms. As it seemed to him that these constructions have some relation with the formation of the lousiness, the shapes of the silk-presses were classified into two types and then the relationship between these types and the lousiness was searched. (The silk-presses have many varieties of shapes.)

(a) **A-type:**

The corpulent part which is composed of a chitin board in the lower part of the silk-press is comparatively thin. This type is seen in the next kinds: Seihaku, Sanmin-san (Silkworms which spin cocoons after third moulting), Chinese No. 17 that has little lousiness, and Kuwako×Kasan.

(b) **B-type:**

The corpulent part which is composed of a chitin board in the lower part of the silk-press is comparatively thick and this type is seen in the next kinds;

European No. 16, Chinese No. 17 that has severe lousiness, Japanese No. 112, and Tegusu san (Silk-gut silkworms).

These shapes are shown in the appendant Fig. 5.

**A. Measurement of the Spinnerets**

The cocoons which the silkworms had just finished to spin were killed by heating, and then made into a skein of 100-reels through a single cocoon reeling. Immediately after that, the silkworms were fixed by Carnoy's solution, and their spinnerets were extracted, cut into a section of  $20\mu$  by the writer's celluloid method,<sup>(4)</sup> and thus the shapes of the spinnerets were examined.

**B. Measurement of the Branched Fibers**

After the various kinds of undegummed silk of different layers were given the required fixation and dyeing, they were cut into sections by the writer's method,<sup>(4)</sup> steeped in the formic acid solution of 80% conc., and swelled at the room temperature; and their branched fibers were measured, as in the appendant Fig. 6 and Fig. 8. In this case, the swelling percentage of the branched fibers is higher than that of the main fibers, and so the observation is easy and accurate. (In the appendant, Fig. 8 G representing the race European No. 18, it is most remarkably perceived.) The dyeing should be different according to the nature of the solution. In this experiment the solution was an acid one and acid fuchsin was used. In swelling a caustic soda solution of 1.5 to 5% conc. was tried, but this solution was found out not to be adequate for observing the branched fibers, for the difference of swelling between sericin and fibroin was large and, besides, sericin started to solve even by a slight change in the temperature.

**C. Measurement of the Lousiness and the Separate Fibers**

Each sample of the skeins was wound on a glass drum 10 cm in width, boiled twice for 40 minutes with 0.5% marceille soap, and dried after the required processing. Then it was removed from the drum, opened up and

examined with a 60 times microscope. The lousiness in the sample was numbered, and the separate fibers are shown by the signs - + # ## ### and so on, and the origination of the lousiness is shown in A, B, C, of the appendant Fig. 7. The following is a sample of measurement.

Table 9.

Kind of cocoon	Layer of cocoon			1st layer			2nd layer			3rd layer			4th layer			5th layer		
	Items	L.S.	S.F.	B.F.	L.S.	S.F.	B.F.	L.S.	S.F.	B.F.	L.S.	S.F.	B.F.	L.S.	S.F.	B.F.		
Seihakn		0	-	0	0	+	0	1	##	0	0	-	0					
Sanmin-san		0	-	0	0	-	0	2	##	0	7	##	14	0	##	0		
Chi. 17 L.* slight		0	-	0	0	+	4	1	+	0	0	+	0	0	-	0		
Kuwako x Kasan		0	-	0	0	-	0	0	-	0	0	-	0					
Eur. 16		0	-	0	0	-	0	0	-	0	0	-	2	0	-	0		
Chi. 4		0	-	0	4	##	20	3	##	10	0	-	5					
Eur. 18		0	-	1	14	##	24	9	##	90	0	-	2					
Chi. 16		0	##	0	14	##	0	8	##	0	0	-	0					
Chi. 17 L.* Severe		0	-	20	1	+	20	7	##	0	0	-	2	7	##	0		
Jap. 112		0	-	1	8	##	20	10	##	10	0	-	0					
Tegusu-San		0	-	0	0	-	0	0	-	0	0	-	0					

Remarks: L.S.....Lousiness-spot. S.F.....Separate fiber.  
 B.F.....Branched fiber.  
 \* L.....Lousiness.

Remarks:

L.S.: Lousiness-spots

Real measurements of lousiness-spots appeared on each of 50 cocoon-fibers which are 10 cm in length.

S.F.: Split fibers

As it was impossible to observe them in their proper state of split fibers, these fibers are also included in them.

B.F.: Branched fibers

Real measurements of them among 50 cocoon-fibers

The fiber lengths of the cocoons used in this experiment are as follows:  
 (Table 10.)

Table 10.

Kind of cocoon	Sei-haku	3 min-san	Chi. 17 L. Slight	Kuwako × Kasan	Eur. 16	Chi. 4	Eur. 13	Chi. 16	Chi. 17 L. Severe	Jap. 112	Tegu-susan
Length of cocoon fiber (kai)	400	650	900	700	800	600	800	600	800	600	600

As seen in the above results, the relation between the number of the lousiness and the branched fibers is not the same with the relation between the number of the lousiness and the separate fibers.

The relations of the lousiness to the shapes of the silk-press mentioned previously are shown in the following table. (Table 11.)

Table 11.

Kind of cocoon	Sei-haku	3 min-san	Chi. 17 L. Slight	Kuwako × Kasan	Eur. 16	Chi. 4	Eur. 18	Chi. 16	Chi. 17 L. Severe	Jap. 112	Tegu-susan
Form of silk-press	A	A	A	A	B	B	B	B	B	B	B
Measure part (from 1st layer)	<sup>m</sup> 337.5	337.5	337.5	337.5	337.5	337.5	337.5	337.5	337.5	337.5	225.0
Branched fiber	Non	very Slight	Non	Non	Non	Exist	Many	Non	Non	Exist	Non
No. of lousiness	very Slight	very Slight	very Slight	Non	Non	Slight	very Severe	very Severe	very Severe	very Severe	Non
No. of Separate fiber	Many	Many	Slight	Non	Non	Many	Many	Many	Exist	Many	Non

## Remarks:

The branched fibers were examined in the state of the cocoon fibers which were cross-sectioned (their thickness  $10\mu$ ) and swelled with the 80% formic acid solution. The observation was made by the use of a 600×microscope. The number of the lousiness was examined of 50 cocoon-fibers, 10cm in length, which were degummed in the boiling state for 80 minutes with the 100 times of the 0.5% marceille soap solution and were dried.

Separate fibers were examined with the same samples as mentioned above by the use of a 60×microscope.

From the above table, it can be considered that the shapes of the silk-presses have some relations with the formation of the lousiness. That is, in Type A, there is a little or no lousiness emerging, but in Type B very much lousiness appears. That these relations are especially perceived in the case of Chinese No. 17 makes the writer feel that the above consideration is true. Thus the writer makes his further investigation in this problem. By the way, the Chinese No. 17 is a valuable material, because it was descended



for several generations by Assistant Professor S. Yamaguchi. The lousiness has closer relation with separate fibers than with branched fibers. The difference in the state between branched fibers and separate fibers makes what the writer calls "the splitting of the main fibroin" more affirmative. In order to make this explanation clear, the photographs are used. From A to F in Fig. 3, one of the appendant figures, shows the disintegrating progress of fibroin; A shows the splitting at very short lengths. B and C show that the splitting of the whole fibroin takes place over its wide range, but it is thought that in such thickness of split fibers as shown by B and C they do not assume the form of lousiness, i.e. they come together only to form a mass. At D the splitting further goes on and the split fibers of about  $1\mu$  in width are perceived. E shows further splitting and in this state they already form the lousiness by themselves. When broken fibers collect together at one place, they show themselves as such big sizes of the lousiness as seen at F.

The lousiness formed by the above-mentioned process is shown in the appendant Fig. 1. When a part of the lousiness is examined, granules are often seen among the fibrils, especially very often in the center of closely packed fibers. These granules are thought to be the amorphous parts or similarities of sericin among the fibril bundles, or the amorphous parts that have not been dissolved because of being surrounded by the fibrils. If they consist of sericin, they must be hardened to some extent after the mass parts have been dried.

But the contrary is the case. Mr. Suzuki<sup>(5)</sup> stated that they are sericin masses, gluey matter. But his opinion is different from the writer's opinion. The writer<sup>(6)</sup> once experimented on the gluey matter and found that the fibroin in it is always bigger in size than the lousiness-forming fibers. Append. Fig. 4 shows the cross-section of the split parts of fibroin. A in the figure represents the splitting which is starting from one end of fibroin with an arrow mark, and B shows some unsplit parts in the center (horse-shoe-shaped) of the fibroin and C shows the complete splitting. This figure shows very plainly the splitting of fibroin.

Append. Fig. 2 represents the real lousiness, showing a collecting process of the fibrils. Append. Fig. 2 shows the lousiness which lies hidden deep in the threads at their mechanical friction and therefore is hard to be found by the naked eyes. Append. Fig. 1 represents the lousiness that appears on the surface of the threads or a cloth, and more than 80% of lousiness belong to this type. Therefore this type in reality does harm to the textiles.

### 5. The Splitting Quality of the Silk Fibers after Swelling Treatment

Observing the splitting quality of silk structurally, it is assumed that this quality is due to the differences in the cohesive power of micells or micell bundles.

If the cohesive power follows Van der Waal's law, the swelling of silk and the enlargement of the inter-micellar space cause changes in cohesion, and at a wider part of the inter-micellar space the gravitation between the molecules becomes small under the same stress and so perhaps there are much more chances of separating micell-bundles.

The following experiments were conducted on this assumption. It is known that formic acid swells silk well. Silk as a sample was swelled with 80% formic acid and, according to circumstances, was pressed between slide glasses and immediately after this degummed by steeping in the degumming solution. The materials consisted of 8 cocoon fibers which were wound on a glass reel of 10 cm. width. The temperature of the solution was 20 to 25°C. The silk fibers were transformed and disintegrated by swelling and pressing. The materials were boiled with the 0.5% soap solution at the temperature of 97 to 98°C. for 80 minutes and once more were subjected to the same treatment. One of the same materials as a control was subjected to the regular degumming. The following is an example of the experiments.

Table 12.

Kind of cocoon	Items Layer of cocoon	Denier of cocoon fiber	Control		Experiments			
			No. of lousi-ness	Sepa-rate fiber	No staining		Staining	
					No. of lousi-ness	Sepa-rate fiber	No. of lousi-ness	Sepa-rate fiber
3 min-san	1st layer	1.9 <sup>d</sup>	0	—	1	—	0	—
	2nd "	2.5	0	—	0	—	0	—
	3rd "	2.3	0	+	2	+	0	+
	4th "	2.2	0	+	0	—	0	—
	5th "	2.2	0	—	0	—	0	—
Seihaku	1st layer	3.2	0	—	0	—	0	—
	2nd "	3.5	0	—	0	—	0	—
	3rd "	3.3	0	—	0	—	0	—
	4th "	3.1	0	—	0	—	0	—
	5th "	2.4	0	—	0	—	0	—

## Remarks:

The observation was made by the use of a 100× microscope.

The dyeing of the degummed silk was done in the solution of the temperature of 40°C. for 60 minutes. Acid soler cyanine as a dyestuff was used for this experiment.

Table 13.

Kinds of cocoon	Items Layer of cocoon	Denier of Cocoon fiber	Control		Experiments			
			No. of lousiness	Sepa- rate fiber	No staining		Staining	
					No. of lousiness	Sepa- rate fiber	No. of lousiness	Sepa- rate fiber
Jap. 115×Chi. 108	1st layer	3.6 <sup>d</sup>	3(small)	≡	2	≡	2	≡
	2nd "	3.4	7	≡	12	≡	4	≡
	3rd "	3.5	8	≡	11	≡	11	≡
	4th "	3.6	8	≡	10	≡	13	≡
	5th "	3.5	23(small)	≡	18	≡	8	≡
Over-matured worm (1)	1st layer	2.8	2(small)	+	6(small)	≡	6	≡
	2nd "	2.8	2(small)	+	6(small)	≡	4	≡
	3rd "	2.2	4	+	8	≡	7	≡
	4th "	1.8	1	+	4	≡	6	≡
	5th "	1.6	0	+	7	≡	4	≡
Over-matured worm	1st layer	3.7	3	≡	8	≡	4	≡
	2nd "	3.2	2	≡	12	≡	13	≡
	3rd "	2.6	3	≡	8	≡	8	≡
	4th "	2.2	1	≡	8	≡	5	≡
	5th "	1.8	0	—	5	≡	0	+

## Remarks:

The over-matured silkworm No. 1 is the one that started cocooning 24 hours after its forced stopping at the beginning of cocooning in its ordinary full-grown state. The over-matured silkworm No. 2 is the one that has been controlled for 48 hours in the same way.

In the test-division, the dyeing-division has less lousinesses than the non-dyeing division. This is because the masses of lousiness fell off during the treatment. In Table 12 and 13, each kind of the silkworms did not show a fixed tendency contrary to the writer's expectation: The Sanmin-san and Seihaku had few separate fibers and little lousiness both in the press-treatment and the control division and almost no difference was seen between the both kind while Japanese No. 115×Chinese No. 108 and the over-matured silkworm division of the same kind increased in the lousiness as well as separate fibers by the press-treatment. These facts show that the above-mentioned three kinds of silkworms are essentially different in the structures

of their silk, and it can be affirmed that the former fact shows that the silk of Sanmin-san and Seihaku is hard to split and the latter fact shows that the silk fibers of Jap. No. 115×Chin. No. 108 is easy to split.

There are already many reports that Sanmin-san and Seihaku have little lousiness. The writer thinks that the differences in the structure of silk are chiefly the ones in the orientation of the micells: that is, the fibers of the former kind are disorderly in the orientation of micells, which fact means the incomplete stretching in the spinning of silk and also that the fibers are difficult to split because the fibrils are not formed in parallel, while the fibers of the latter kind are orderly and parallel in the orientation and therefore the micell-bundles in parallel arrangement becomes easily separated by swelling and pressing. It is structurally because of the great splitting quality that splitting takes place by the ordinary degumming. In this case, splitting takes place not only in the main fibers, but also in the branched fibers in the cocoon layers which are produced by the divergence of fibroin in the silk-gland. Structurally, the splitting quality is great in the branched fibers, and therefore there must be much emergence of the lousiness caused by the branched fibers. Much of the lousiness of this origin is so small in the size of its spots that they can hardly be seen by the naked eyes. By the above experiments, it was found out that the disorderly orientation of the micells causes no splitting of the fibers, even though the inter-micellar spaces are enlarged by swelling. Therefore, it can be said that the only orderly orientation of the micells in the formation of silk causes splitting of the fibers by such treatment as degumming.

### III. The Swelling Properties of the Silk Fibers in Various Cocoon-Layers

By the already-mentioned experiments, it was pointed out that the orderly orientation of micells tends to splitting. To make sure this assumption, the degree of the micell orientation was measured as to the silk used in these experiments. P. H. Hermans<sup>(8)</sup> reported that the degree of the micell orientation can be expressed by the ratio between the longitudinal swelling (L) and the lateral swelling (B) of the fiber.

The larger is the ratio, the higher the degree of the orientation will be.

Table 14. B/L in the Third Layer of each Kind of Cocoon.

Kind of cocoon	Longitudinal Swelling (L)	Lateral Swelling (B)	B/L × 100	Degree of lousiness
Seihaku	105.0 <sup>%</sup>	159.6 <sup>%</sup>	152.0	None
Sanmin-san	101.7	167.6	164.9	Severe
Eur. 16	105.8	161.2	152.5	Very little
Chi. 4	100.7	154.4	153.4	Severe
Chi. 16	105.2	151.1	143.7	None
Eur. 18	100.8	205.0	199.5	Very severe
Chi. 17 L. slight	106.9	164.4	153.9	Slight
Chi. 17 L. severe	98.1	196.3	199.9	Very severe
Jap. 112	99.5	169.7	170.5	Severe
Tegusu-san	110.3	172.8	155.6	None

Remarks: Measured at room temp. 20-23.°C, with 80% H. COOH.

B=Cross sectional area

Length of piece for test, taken 7-50  $\mu$ ,

By this experiment, the writer could find out that there exists a close relation between B/L and the emergence of the lousiness. The following table shows the results of the swelling degrees of different cocoon-layers of the kinds shown in Table 12 and 13.

Table 15. Longitudinal and Lateral Swelling of Silk Fibron.

Cocoon layer	Kind of cocoon Item	Jap. 115 × Chi. 108 (1)	Jap. 115 × Chi. 108 (2)	Seihaku	Sanmin-san
		<sup>%</sup>	<sup>%</sup>	<sup>%</sup>	<sup>%</sup>
First layer	Lateral swelling (B)	162.4	160.0	144.0	122.0
	Longitudinal swelling (L)	103.9	103.3	105.0	107.5
	$\frac{B}{L} \times 100$	157	155	137	113
Second layer	Lateral swelling (B)	183.6	171.5	151.0	144.0
	Longitudinal swelling (L)	102.2	102.0	103.7	103.3
	$\frac{B}{L} \times 100$	178.5	168.3	145.0	139.5
Third layer	Lateral swelling (B)	162.3	166.5	154.0	112.2
	Longitudinal swelling (L)	102.5	103.4	103.0	103.0
	$\frac{B}{L} \times 100$	158.0	161.0	149.0	108.5

Cocoon layer	Kind of cocoon	Jap. 115 × Chi. 108 (1)	Jap. 115 × Chi. 108 (2)	Seihaku	Sanmin-san
	Item				
Forth layer	Lateral Swelling (B)	152.6	167.0	144.5	110.5
	Longitudinal swelling (L)	103.9	106.5	102.5	104.8
	$\frac{B}{L} \times 100$	147.0	156.0	140.0	105.0
Fifth layer	Lateral swelling (B)	143.3	150.3	126.0	110.0
	Longitudinal swelling (L)	101.4	104.4	100.7	105.2
	$\frac{B}{L} \times 100$	140.8	143.0	124.0	104.7
Average	Lateral swelling (B)	160.8	165.3	143.9	119.7
	Longitudinal swelling (L)	102.8	103.9	103.0	104.8
	$\frac{B}{L} \times 100$	156.3	156.7	139.0	114.1

Remarks: (1) Normal matured silkworm.  
(2) Over-matured silkworm.

As shown in Table 15, the ratio is large in the races abundant in lousiness and also is high in the middle-layer of cocoon-layers of any race of silkworms which fact accords entirely with the writer's assumption. Judging from the results of these numerous experiments, it can truly be affirmed that the fundamental cause of the emergence of the lousiness is essentially due to the condition in the formation of silk, and it is only a superficial view that the cause consists merely in the branched fibers as the views of numerous researchers hitherto. The following experiment was conducted for the purpose of seeing whether only the branched fibers in the cocoon-layers make themselves the fibers which form the lousiness and then form the masses.

The 8 threads of the cocoon fibers from over-matured silkworm cocoons of each of the three kinds Sanmin-san, Seihaku and Japanese No. 115 × Chinese No. 108 were wound on glass-reels, 10 cm in width.

These samples were steeped in the 5% NaOH solution contained in Petri's dishes, 16 cm. in diameter, in which the samples were kept in the depth of 0.5 cm., the temperature of the solution being 15 C. Then the separate fibrils were observed with a 60× magnifying glass 10 minutes after the steeping. Next, the samples were taken out of the Petri's dishes and were observed after washing and drying. The writer called the method of the former observation the steeping division and the method of the latter

observation the drying division. Next, the samples were coloured with the 0.1% Soler cyanin solution having pH of about 4.5 at the temperature of from 15 to 18°C for 3 hours and then were observed. He called this method of observation the dyeing division. As a control the same samples were subjected to the ordinary method of degumming and dried and then observed.

He called this the degumming and drying division. Next, these samples (ordinarily degummed samples) were dyed at a low temperature and were observed. He called this the degumming dyeing division.

Table 16. Emergence of Branched Fibers, Lousiness and Separate Fibers After Treatments.

Kind of cocoon	Item Cocoon layer	5% of NaOH						Control			
		After steeping		After drying		After dyeing		After drying		After dyeing	
		B.f.	L.s.	B.f.	L.s.	B.f.	L.s.	B.f.	L.s.	B.f.	L.s.
Samuh-san	First	-	0	-	0	-	0	-	0	+	0
	Second	+	0	+	0	+	0	+	0	+	0
	Third	≠	0	≠	0	≠	0	≠	0	≠	3 (small)
	Forth	-	0	≠	0	+	0	+	0	+	0
	Fifth	-	0	≠	0	≠	0	+	0	+	0
Seihaku	First	+	0	-	0	+	0	+	1	+	2
	Second	-	0	-	0	-	0	-	0	-	0
	Third	-	0	-	0	+	0	-	0	-	0
	Forth	-	0	+	0	+	0	-	0	-	0
	Fifth	-	0	-	0	+	0	-	0	-	0
Jap. 115× Chi 108 over- matured	First	≠	1	≠	2 (small)	≠	2 (small)	≠	20	≠	20
	Second	≠	0	≠	0	≠	0	≠	12	≠	12
	Third	≠	0	≠	0	≠	0	≠	7	≠	7

Remarks: Few branch fibers in the treatment of 5% NaOH, are long.  
Degree of lousiness increases by high temperatura dyeing.

In this table, no separate fibers and lousiness are found both in the 5% NaOH and the control divisions of the race Seihaku whose cocoon fibers have no branched fibers and have the weak splitting quality. But among the divisions of the over-matured silkworms the cocoon layers of which have branched fibers, the 5% NaOH division produces many branched fibers and no lousiness. The control has a great number of branched fibers and much lousiness.

From these results, it may be assumed that the branched or separate fibers do not form the lousiness by themselves, but, after these fine fibers have been formed, the action of aggregating them takes place and thus masses of the fine fibers are formed.

#### IV. The Dyeing Properties of the Main Fibers and the Split Fibrils

It is an established theory that the masses of the lousiness have lower absorptiveness toward dyestuffs than the main fibers and therefore they are dyed pale. Dr. Ohara<sup>(9)</sup> points out that the cause is the difference in pH between the two, but has not determined it.

He also describes that the difference takes place remarkably in the degumming process. The following experiments were conducted to see if there was any difference in the absorptiveness of the dyestuffs between the two.

The kind European No. 19×Chinese No. 17 as a sample was subjected to the regular degumming and then the masses of the lousiness that appeared were carefully gathered together using a pair of pincettes. Two types of samples were made, one free from the lousiness to be seen by the naked eyes and the other containing much lousiness. But microscopically, the former contains small sizes of the masses of lousiness and branched fibers, and the latter contains separate fibers and a few main fibers.

The percentage of mixture of the lousiness and the main fibers in the two was examined as follows: (Table 17, 18 and 19)

After the test of the absorption of the dyestuffs both of them were treated with the G-solution. After this treatment, dissolved parts were regarded as the ones of the fibrils and lousiness and the remained parts were thought as the main fibers.

Thus the ratios in the two samples were examined.

Table 17. First Test.

Classification	Main fiber		Lousiness	
	wt. of adsorption	% of adsorption	wt. of adsorption	% of adsorption
Solar cyanin	0.6216 mg.	99.45 %	0.6181 mg.	98.89 %
Direct brown	0.590	94.40	0.6146	98.33
Rhodamin B conc.	0.5591	89.49	0.5515	83.24

Remarks: Residue of dyeing solution showed no color difference except basic-dye.



Table 18. Second Test.

Classification	Main fiber		Lousiness	
	wt. of adsorption	% of adsorption	wt. of adsorption	% of adsorption
Soler cyanin	1.0149 mg.	97.58 %	0.9776 mg.	94.0 %
Direct brown	0.6858	65.94	0.7082	68.10
Rhodamin B conc.	0.8855	85.14	0.7894	75.90

Table 19. Percentage of Main Fiber and Fine Fiber in each Sample.

Classification	Sample	wt. of Sample	After treatment in G-sol <sup>a</sup>	Percentage of fiber	
				Main fiber	Fine fiber
Acid dye	Main fiber	46.8 mg.	38.5 mg.	82.26 %	17.74 %
	Lousiness	44.8	22.6	50.44	49.56
Direct dye	Main fiber	48.6	40.7	83.74	16.26
	Lousiness	46.0	27.4	59.56	40.44
Basic dye	Main fiber	47.0	38.0	80.00	20.00
	Lousiness	44.5	23.4	52.58	47.42

Remarks: The G-solution treatment was conducted at the temperature of 50°C for 5 minutes.

As seen above, regarding the affinity for dyestuffs, the division of the main fibers showed the higher affinity for acid or basic dyes, but the lousiness division showed the higher affinity for direct dyes.

The following quantitative difference in the absorptiveness based on the experiments is required to make the difference in colour visible. As a sample, the lousiness-removed fibers were used.

Dyestuff	Pale colour part	Heavy colour part	Difference
Acid dye	0.62%	0.92%	0.30%
Direct dye	0.23	0.482	0.252
Basic dye	0.28	0.556	0.275
Time of dyeing (m)	3	15~18	12~15

This colouring difference is plainly discriminated by the naked eyes, but is much weaker than that in the lousiness.

If the diameters of the fibers are same and there is any difference only

in absorbing amount, there appears no colouring difference making the lousiness visible to the naked eye, even if there is the difference of 1 to 2. The silk fibers producing the lousiness contains a great number of fine fibers which do not show themselves as the masses and have almost the same diameters in thickness with the parts of the masses. These fine fibers do not show the colouring difference to the naked eye. As mentioned before in the lousiness division of the direct dyestuff divisions, the masses of the lousiness are observed to be in the light hue in spite of their high absorptiveness, and show the same colouring difference as in the acid and basic dyestuff division. From these facts, only the differences in thickness of and in properties of the fibers are not sufficient to account for the light hue of the lousiness division. Accordingly, the writer pointed out, as the causes of the light hue of the lousiness division, (1) the difference in the dyestuff absorptiveness coming from the structural differences in the main fibers and separate fine fibers, (2) the difference in the speed of the dyestuff absorption, and (3) the difference in light-transmissibility coming from the differences in thickness of both of the fibers.

## V. The Experimental Observations on the Origination of the Lousiness

By the various experiments set forth in the sections from II to IV, it was affirmed that the lousiness is formed by the fine splitting of silk fibers. And it was also pointed out that this splitting happens in the branched fibers as well as in the main fibers. This section deals with the causes of the formation of the branched fibers and split fibers, the splitting quality viewed from the swelling properties of the main fibers and branched fibers, and the experiments on the process of formation of the masses of the fine fibers produced by these causes.

### 1. The Formation of the Branched Fibers and Split Fibers

#### A. Branched Fibers

There have been numerous researchers who regarded branched fibers as the cause of the formation of the lousiness, and recent studies in Japan have been devoted exclusively to this field, as seen in the reports of Takami,<sup>(10)</sup> Kikkawa, Muroga,<sup>(11)</sup> and Ōba.<sup>(12)</sup> They have stated that the plait-like forma-

tions on the surface of fibroin in the silk-gland are the cause of the formation of branched fibers. The writer agrees with the above researchers on the presence of the plaits, but does not agree to Takami's opinion that this is the only cause of the formation of branched fibers. He does not agree to any of these researchers' opinions that branched fibers by themselves form the lousiness. As the causes of the plait-like formations the writer would like to point out these: the difference in the maturing degrees of the fibroin substance between the central portion and the outer portion of fibroin in the silk-gland, and the extrusion caused by the continual movement of the silk substance from the posterior gland, with an increase in the amount of silk secreted. The shapes of the plait-like formations are shown in A and A' of append. Fig. 10, B and C of Fig. 11, and Fig. 12. Takami denies the presence of the separate linear fibroin in the silk-gland and states that it does not become branched fibers, but the writer<sup>(13)</sup> perceives the separate linear fibroin in the silk-gland and cannot help thinking that the fine fibers adhering in linear form to the surface of the main fiber after the degumming of the fixed silk-gland are produced from the fibroin separated from the sericin layer of the silk-gland, as seen in D and E of append. Fig. 13 and in J, K, D' and K' of Fig. 14. On such a structure as of free linear fibroin Dr. Nakata<sup>(14)</sup> has reported, and Jun-ichi Suzuki<sup>(15)</sup> has reported on free granular fibroin. Takami states that there exist in the body of a live silkworm the spiral and serpentine shapes of fibroin which the writer perceived in the parts of the middle and anterior silk-gland after given a clairvoyant treatment, but if there were these shapes in the silk-gland, the silk spinning of the silkworm in cocooning would have to be performed only by stretching. The writer, however, has never seen such shapes in the liquid silk in the silk-gland as extracted from the body of the live silkworm even once in his many observations of thousands of silkworms. These shapes can be seen in this way: when observed with a microscope, the liquid silk in the silk-gland comes to assume the shapes already mentioned by taking a spiral or extending motion according to the differences in the properties of soaking solutions. Their emergence takes either a short time or a long time. For example, in case the pH of soaking solution is 9 to 10, the spiral motion starts within 30 seconds and stops after 30 to 60 seconds. Accordingly, if observed after this time, the contents of the silk-gland become entirely stationary and fix themselves into certain shapes and thus these shapes can be observed as if they were natural in the silk-gland. The liquid silk in the middle-gland\* is easily deformed when immersed in solutions of various pH. For example, within the

pH 3.6 to pH 10 a remarkable transformation was observed. If such a transformation takes place in the gland tunnel of a limited space, the fibroin swells in the direction of the gland axis and thus assumes serpentine and spiral shapes. The maturing degree of the fibroin in this part of the silk-gland can be considered to be considerably advanced. The natural extension of the liquid silk was measured of the thickest part of the middle-gland, as follows:

Table 20. Natural Extension of Liquid Silk in Middle Gland when steeped in 0.01% CH<sub>3</sub>COOH, at Room Temp., 25°C.

Kind of cocoon	Length of sample	Time of treatment						
		min. 0	min. 2	min. 5	min. 10	min. 15	min. 20	min. 30
Jap. 115 × Chi. 108	Length of sample	2.9 cm.	3.3	3.7	4.45	4.9	4.91	4.90
	Elongation	100%	113.7	127.5	153.4	168.9	169.	168.9
Jap. 112 × Chi. 110	Length of sample	3.2 cm.	3.5	3.95	4.5	4.75	4.8	4.8
	Elongation	100%	109.3	123.4	140.6	148.5	150	150

As seen in Table 20, the extension percentage ranges from 150% to 170%. If the liquid silk is freed from sericin by degumming with the 0.5% soap solution for 80 minutes after being steeped in 1% acetic acid of pH 2.8, the spaces between the plaits of extended silk are enlarged and there is no change in the number of plaits in case the original sample has plaits.

\*... To shows middle division in the silk gland, those that follow are the same.

### B. The Split Fibers

The split fibers refer to the silk fibers splitted by various processes. They, therefore, are varied in thickness. They include the split branched fibers and the split main fibers and, in fact, there is no distinction between both fibers. If the lousiness has its origin in the branched fibers as some researchers have said, the next case is inexplicable: as the cause of formation of the branched fibers is definite in the gland, the number of the branched fibers in a cocoon layer is fixed and so the number of the spots of the lousiness must also be fixed. There are, however, some differences in the number of the spots of the lousiness between the fibers of the extremely over-matured silkworm's cocoon which produces much lousiness, those of the first spiracle-closed silkworm's cocoon which produces much lousiness, and those of the cocoon spun at such extremely high temperatures at which the lousiness decreases in its appearance as seen in the second report. If the silk substance

in the silk-gland without plait-like parts is first stretched to the coagulation increasing point (C.I.P.) and is pressed, rubbed, and then degummed, it produces a great number of split fibers and much lousiness. If a silk-gland is extended under proper stretching conditions, it is transparent at first but becomes opaque suddenly just before reaching the C.I.P., changing to be of optical inequality. But when this coagulation is dried, it becomes transparent again. The reason why it becomes opaque is that the moisture and other amorphous matters accumulate between the micells or chain-bundles of micells while the fibroin micells are orientated and therefore there happens the difference in their refractivity. The fibroin micells form conglomerate bundles indefinite in number and there are produced various sizes of spaces in the micells.

If the fibroin is strongly extended beyond the increasing point (C.I.P.), the micells that have been paralleled earlier begin to break longitudinally, while the ones whose orientation has been delayed are gradually orientated. But the fiber as a whole does not break down. In this state, the micell bundles start a sliding motion, sliding among the adjacent micells. This fact proves that, in stretching the liquid silk, a stress-strain diagram takes a smooth curve up to the C.I.P., but, beyond this, takes a staircase shape.

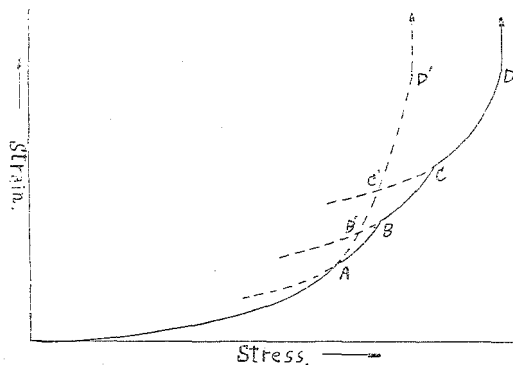


Fig. 2.

In the Fig. 2, the C.I.P. is marked A. If stretching is stopped and stabilized at this point, the strength of the fiber becomes the maximum.

If extension continues, the curve runs in the direction of B, C and D and at the point D occurs a break-down. The writer assumes that this is because the orientation of some micells in the fiber becomes maximum at the

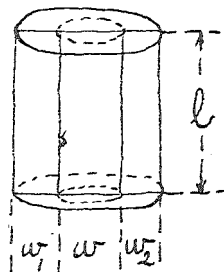
points A and B, and the subsequent extension brings about its longitudinal break-down. The writer<sup>(17)</sup> has known by his experiment that the strength of the fiber made by stretching the silk-gland of an over-matured silkworm up to the C.I.P. becomes smaller and also that the strength of the fiber made by stretching it beyond the C.I.P. becomes still smaller. If raw silk or cocoon fibers are cut off by stretching, laceration or splitting is often perceived at the cut-end, which phenomenon can be explained by the above account. Append. Fig. 15 shows the section of a bundle of micells, and Fig. 16 shows a model of the silk fibers broken down.

## 2. The Swelling Properties of the Branched Fibers and the Main Fibers

In the Section III, the writer described clearly that the splitting quality of silk has close relations with its swelling degree. This swelling degree has also much to do with the micell orientation. He pointed out that the maturing degree of liquid silk in a silk gland differs from the outside to the center of the gland. Therefore, it is thought that there is some difference between the swelling degrees of these parts of the coagulated silk-gland. In this experiment, the silk-gland was coagulated and swelling degree of both parts were measured according to the following sampling.

Table 21. Swelling of Fibroin Substance in the Middle Silk Gland.

Classification		Sample	Degree of swelling	
			After 20 min.	After 50 min.
I	Length	1030 $\mu$	1400 $\mu$	1500 $\mu$
	Degree of swelling		139.8%	145.3%
w	Width	1080	1770 $\mu$	1790 $\mu$
	Degree of swelling		163.7%	165.5%
w <sub>1</sub>	Width	200	400 $\mu$	420 $\mu$
	Degree of swelling		200%	210%
w <sub>2</sub>	Width	160	350 $\mu$	390 $\mu$
	Degree of swelling		218.7%	243.7%

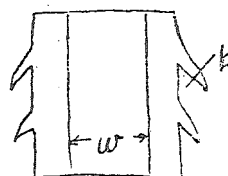


As seen in this table, the outside has a high swelling degree and the center has a low one. If the silk of such different maturing degrees are spun

under the same conditions, the outside has a good orientation of micells and is fine, while the center has a bad orientation of micells, and is rough. The swelling degree of each part of the coagulated silk as shown in the next figure was measured and the results are as follows: (Table 22.)

Table 22. Swelling of Inner Layer of Fibroin and of Branched Part of Fibroin Substance in the Middle Gland

Class.	Width of sample	Degree of swelling	% of swelling
w	600 $\mu$	888 $\mu$	146.6%
b	5.0	10.0	200.



As seen above, it has become apparent that there is a structural difference between the center and the outside of the fibroin in the gland and also that the plait-like part has a different structure.

The swelling degree of the branched fibers existing in a cocoon fiber was measured:

Table 23. Lateral Swelling (in dia.) of Main Fiber and Branched Fiber.

Sample	Main fiber			Branched fiber		
	Dry	wet	%	Dry	Wet	%
1	15.6 $\mu$	19.8 $\mu$	126.7	2.6 $\mu$	4.55 $\mu$	155.0
2	19.8	22.1	114.0	6.5	9.1	140.0
3	16.9	19.8	117.0	2.6	3.25	125.0
4	19.8	26.0	131.0	1.3	1.83	140.0
Average			122.1			140.0
Max.			131.0			155.0
Min.			114.0			125.0

Table 24. Lateral Swelling (in area) of Main Fiber and Branched Fiber.

Sample	Main fiber			Branched fiber		
	Dry	Wet	%	Dry	Wet	%
1	88 <sup>μ<sup>2</sup></sup>	175 <sup>μ<sup>2</sup></sup>	198.8	3.5 <sup>μ<sup>2</sup></sup>	11.0	311
	72	175	227.7	—	—	—
2	88	170	133.4	3.5	13.0	311
	69	142	205.7	2.5	5.0	200
3	113	186	164.5	2.5	8.0	320
	96	140	161.4	3.5	11.0	311
4	139	254	182.7	3.5	10.0	208
	130	228	175.3	—	—	—
Average			188.7			276.8
Max.			213.3			320.0
Min.			162.7			208.0

## Remarks:

Kind of cocoons: European No. 18  
 Steeping solution used: 80% HCOOH  
 Room temperature: 27-28°C  
 Measured with a 860×microscope

The results show that there are some differences between the swelling property of the main fibers and that of the branched fibers, and this fact shows that there is a structural difference between the two by whatever cause the branched fibers may be formed. The high degrees of the micell orientations of the branched fiber and of the outside of the main fiber account that the splitting qualities of these parts are higher than that degree of the center of the main fiber. The degree of micell orientation becomes higher with the multiplication of the stretching of the liquid silk, and shows a sharp elevation if it is extended to the C. I. P. Append. Fig. 17 shows the splitting of the degummed coagulated silk which has been subjected to different multiples of stretching. A in Fig. 17 shows the fibers made by the uniform stretching of silk, and C and D in this Fig. show the fibers stretched up to the C.I.P. in this state, revealing the one-side splitting of fibroin caused by degumming. B in it shows the fibers stretched rather uniformly. If they are stretched up to the C.I.P. in this uniform state, the micell orientation becomes uniform and the fibroin splitting takes place on the whole. E



in this Fig. shows this state. It can be easily thought that also in the case of the silkworm's spinning the fiber of such a structure is formed. Thus there arise the differences in the splitting qualities of fibroin.

### 3. The Formation of Lousiness-spots

When the main fibers or branched fibers are split into fine fibers and then these fine fibers come together into masses, there appears the lousiness. And the writer tried the experiments on the process of the formation of these masses. Cocoon fibers as a sample were wound on a small frame 5 cm in width. They were put in petri's dishes of 1.5% NaOH so that the threads might float in the solution (the temperature of which was 30°C). While they were examined with a 100× microscope, the fine fibers separated from the main fibers increased in length and curved at first as seen in append. Fig. 18 but, when cut at a point, these fine fibers sharply spiraled and curved toward the unseparated point of the main fibers, and finally shrank, as shown in append. Fig. 19. Regarding the time to start the deformation, the finer the separate fibers are, the earlier the deformation begins. For instance, its beginning took 60 to 90 seconds (at the solution-temperature of 25 to 27°C) in the case of the separate fibers of about 1  $\mu$  in diameter. The deformation becomes difficult if the fibers are covered with the deck-glass on the slide-glass. As the result of the deformation, there arise such shapes as spiral or wave-like, as shown in append. Fig. 19 and Fig. 20. The former Shape was observed like this: the diameters of the rings, 28—120=40—80  $\mu$ ; the number of spiral, 2—16; in case the thickness of the separate fibers are 1—1.5  $\mu$ . The latter shape was observed: The wave length, 40—80  $\mu$ ; the wave width, 60—80  $\mu$ . These figures differ according to the concentration of the solution and the conditions of the separation.

The following are samples of the experiments.

Table 26. Dia. of Fiber Causing Deformation of Split Fine Fiber. (1)

Thickness of split fiber		Thickness of main fiber
Spiral fiber	Straight fiber	
1.0 $\mu$	3.0 $\mu$	16.5 $\mu$
1.0	2.5	16.5
0.8	2.5	16.5
1.0	2.0	16.5

Remarks:

Steeped in 1.5% NaOH.

Table 26. Dia. of Fiber Causing Deformation of Split Fine Fiber. (2)

Thickness of split fiber		Thickness of main fiber
Spiral fiber	Straight fiber	
3.0 <sup>μ</sup>	5.0 <sup>μ</sup>	18.0 <sup>μ</sup>
1.0	1.0	15.0
1.0	5.0	18.0
2.5	3.5	15.0

Remarks:

Steeped in 3% NaOH.

Table 27. Dia. of Fiber Causing Deformation of Split Fine Fiber. (3)

Thickness of split fiber		Thickness of main fiber
Spiral fiber	Straight fiber	
1.3 <sup>μ</sup>	2.3 <sup>μ</sup>	14.3 <sup>μ</sup>
2.6	2.5	15.6
1.5	3.0	10.4
1.5	3.25	10.0
2.0	3.0	10.0

Remarks:

Steeped in 3% NaOH.

From the above results, it was found out that there is a limit in diameter for the fibers to start the deformation and the deformation occurs only in the fibers which break down in a short time after their separation. And it was often seen that the fibers that do not deform in a solution of low temperature or in a stationary solution start the deformation and become entangled in the main fibers if the solution temperature is elevated or the solution is stirred. Append. Fig. 21 shows this state.

The mechanism of this deformation may be this: the splitting of the fibers steeped in the solution take place from their surface, while the separate fibers give birth to swelling differences on the surface immediately after the separation, and start to curve toward their sides of lower swelling degrees. Append. Fig. 22 shows the model. As seen in Tables 21 and 22, that the swelling degree is different even in every part of one fiber is also the cause of the curvature of the separate fibers. This is because such a structure as seen in A of append. Fig. 17, is formed when the fiber is formed. It is an interesting matter that the deformation caused by the elevation of the solution temperature and also by stirring the solution has some connection with so-called "Brown's motion" taking place in small particles.

The appearance of the Brown's motion has much to do with the size of a particle; it begins to appear at the diameter of 3 to 5  $\mu$ , and the smaller the particle is, the active the motion is. According to Mr. Winner, it is 2.5  $\mu/S^2$  in the case of the diameter of 1.3  $\mu$ , and is 3.3  $\mu/S^2$  in the case of the diameter of 0.9  $\mu$ . That is, the motion takes place very actively around 1  $\mu$ .

## VI. The Theoretical Consideration on the Origination of Lousiness

Liquid silk in a silk gland at the stage of a full-grown silkworm is the concentrated colloidal solution of 33 to 42% of apparent concentration, and the polymerization degree of the silk substance at the early stage of secretion is low. When such liquid silk is spun, at the stage of a full-grown silkworm, through an orifice of the spinneret, or when a silk-gland is extracted and stretched under proper conditions, the orientation degree of the silk molecules varies according to the following factors:—the maturing degree of the liquid silk, the rate of stretching, the multiple of stretching and the pressure of silk-press. Therefore, various silk in structure and property can be obtained by varying these factors. The changes in the moisture contents and shapes of the silk substance taking place between the stretching of the silk gland and its solidification and stabilization were investigated, and the results are as follows: (Table 28, 29)

As seen in these tables, the swelling degree of the liquid silk immediately after being extracted or of the silk immediately after being stretched is 350 to 400% and the proportion of the fiber to the moisture in volume is assumed to be approximately 1:3. If the normal cocoon fiber<sup>(18)</sup> is swelled in the water of about pH. 7.0 119% in width for the outer layer, 133.5% for the middle layer and 124% for the inner layer can be obtained.

From the results of the previous tables, these swelling degrees indicate around 50% of the moisture contents. Next, let the writer consider how the fiber is formed under such stretching. In the non-stretching division, it is assumed that particles of the silk at a high degree of swelling are dispersed at random in free directions in water without orientation. If such liquid silk is stretched, the chain molecules start orientation sliding in the direction of stretching, combine with each other, and increase in the length of the chain, and the adjacent molecules cohere forming their hydrogen bonds sideways. At this time, the excess moisture flows and coheres together.

Table 23. Deformation of Stretched Liquid Silk.  
(untreated)

Stretched	Items	Time elapsing after stretching	After Stretching	Time elapsing after stretching					True cross sectional area of samples	
				5 min.	15 min.	30 min.	40 min.	60 min.		120 min.
5 ×	Swelling	Area of fiber	0.385mm <sup>2</sup>	0.196	0.1385	0.1255	0.1195	0.114	0.08831mm <sup>2</sup>	
		Degree of swell.	337%	171.5	121.5	111.0	104.5	0		
		True area of fiber (%)	22.93%	45.05	63.76	70.36	73.89	77.47		
	Moisture	wt. of moisture	0.049 g	0.037	0.029	0.018	0.011	0.002		0
		% of moisture	64.5%	58.64	51.33	39.56	28.57	6.78		0
10 ×	Swelling	Area of fiber	0.501mm <sup>2</sup>	0.332	0.196	0.159	0.132		0.10226mm <sup>2</sup>	
		Degree of swell.	379%	251.5	148.5	120.5	0			
		True area of fiber (%)	20.4%	30.80	52.17	65.55	77.47			
	Moisture	wt. of moisture	0.045 g	0.0395	0.0265	0.0145	0.008	0.0015		0
		% of moisture	65.0%	61.71	51.96	37.17	24.24	5.76		0
15 ×	Swelling	Area of fiber	0.1375mm <sup>2</sup>	0.091	0.0813	0.0418	0.0382		0.02959mm <sup>2</sup>	
		Degree of swell.	360.0%	23.80	213.0	109.3	0			
		True area of fiber (%)	21.51%	32.52	36.39	70.78	77.46			
	Moisture	wt. of moisture	0.0406 g	0.031	0.016	0.004	0.014	0		
		% of moisture	78.07%	68.88	53.33	23.22	9.00	0		
35 × (Max. stretch)	Swelling	Area of fiber	0.283mm <sup>2</sup>	0.1075	0.085	0.076	0.0713		0.05523mm <sup>2</sup>	
		Degree of swell.	397.0%	151.3	119.0	106.6	0			
		True area of fiber (%)	19.51%	51.37	64.97	72.67	76.39			
	Moisture	wt. of moisture	0.0517 g	0.0337	0.0097	0.0027	0			
		% of moisture	65.45%	55.24	26.24	9.6	0			

Remarks: True area of fiber is obtained as follows:

$$\text{True area of fiber} = \text{Sectional area} \times \frac{1.149}{1.483}$$

1.149=S.G. Obtained from Sectional area,

1.483=S.G. Measured in CH<sub>3</sub>OH absolute,

$$\text{Degree of swelling} = \frac{\text{Sectional area of fiber in each instant}}{\text{Sectional area of fiber in dry state}} \times 100$$

$$\% \text{ of true area} = \frac{\text{True area}}{\text{Sectional area of fiber in each instant}} \times 100$$

$$\text{Percentage of Moisture} = \frac{\text{Moisture content of sample}}{\text{Weight of sample in air}} \times 100$$

Table 29. Deformation of Stretched Liquid Silk,  
(Treated in 1% CH<sub>3</sub>COOH, 30 sec. steeped)

Stretch- ed	Items	Time elapsing after stretch- ing	After Stretching	Time elapsing after stretching						True cross sectional area of samples
				5 min.	12 min.	20 min.	30 min.	40 min.	60 min.	
5 ×	Swelling	Area of fiber	0.753mm <sup>2</sup>	0.440	0.283		0.238		0.221	0.1712mm <sup>2</sup>
		Degree of swell.	341.0%	199.1	127.7		109.5		0	
		True area of fiber (%)	22.73%	38.90	60.49		71.93		77.46	
	Mois- ture	wt. of moisture	0.0745 g	0.0585	0.0415		0.0235	0.0065	0	
		% of moisture	68.34%	62.90	54.6		40.51	15.85	0	
10 ×	Swelling	Area of fiber	0.385mm <sup>2</sup>	0.238	0.181		0.1255		0.114	0.08831mm <sup>2</sup>
		Degree of swell.	337.0%	208.5	158.6		110.1		0	
		True area of fiber (%)	22.93%	37.10	48.79		70.36		77.46	
	Mois- ture	wt. of moisture	0.053 g	0.036	0.021		0.006	0		
		% of moisture	72.6%	62.06	48.83		21.42	0		
15 ×	Swelling	Area of fiber	0.476mm <sup>2</sup>	0.238	—	0.1555	0.1382	—		0.1071mm <sup>3</sup>
		Degree of swell.	344.1%	172.5	—	112.6				
		True area of fiber (%)	22.5%	45.0	—	68.88	77.49	0		
	Mois- ture	wt. of moisture	0.0337 g	0.024	0.012	—	0.003	0		
		% of moisture	69.19%	61.53	44.44	—	16.66	0		
35 × (Max. stretch)	Swelling	Area of fiber	0.43mm <sup>2</sup>	0.196	—	0.124	0.106			0.0821mm <sup>2</sup>
		Degree of swell.	405.0%	185.0	—	117.0	0			
		True area of fiber (%)	19.09%	41.88	—	66.20	77.45			
	Mois- ture	wt. of moisture	0.0575 g	0.046	—	0.021	0.011	0.005	0	
		% of moisture	69.27%	64.33	—	45.16	30.13	9.23	0	

Next, volume contraction with evaporation of moisture of extracted silk-gland is shown in the following graph.

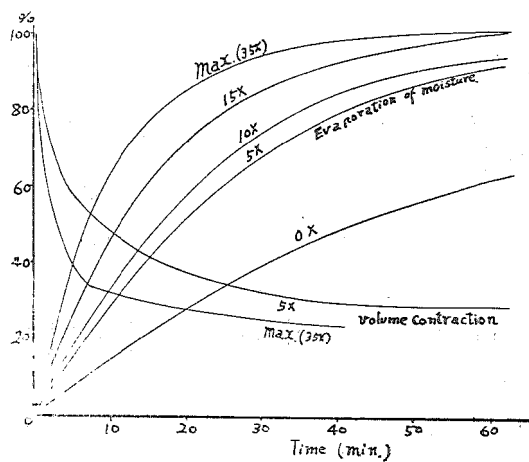


Fig. 3. (Untreated)

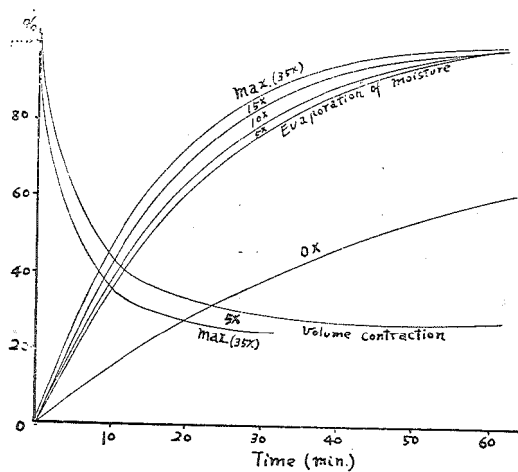


Fig. 4. (Treated)

Because its evaporation does not follow the stretching and solidification of the fiber, the moisture is discharged in the liquid state out of the micells. As long as the multiple of stretching is small, the direction of the molecular chain of the silk forms a certain angle with the direction of the fiber axis, statistically, and the combination is at random. The inter-miceller spaces at this time are rather uniform and large. If the stretching is elevated up to the coagulation increasing point, the chain almost completely forms the parallel orientation to the fiber axis. At this time the excess moisture that gathers outside the micells is pressed out of the micell chain-bundles and gathers together, and thus the inter-miceller space of this part is widened. Therefore inter-miceller spaces and micell chain-bundles spaces are produced in the fiber formed; the former are small and the latter are large. If, as to the moisture exhalation in the fiber thus formed, the comparison is made between the non-stretching division or the division of a small multiple of stretching in both of which the chains are arranged at random and the division of a large multiple of stretching, it is assumed that the latter is always higher in the moisture exhalation than the former. The velocity of the moisture exhalation as shown in the previous tables accords very well with this assumption.

Concerning the shrinkage of the volume of the liquid silk, the results of the tables show that the more the multiple of stretching is increasing, the larger the degree of this shrinkage becomes. This is because the more the degree of stretching increases, the closer the distances between the chains become, and the shrinkage in the chain-bundles is strongly brought about by the exhalation of the moisture. The shrinkage of the fibers in the course of drying is shown in the following model figures.

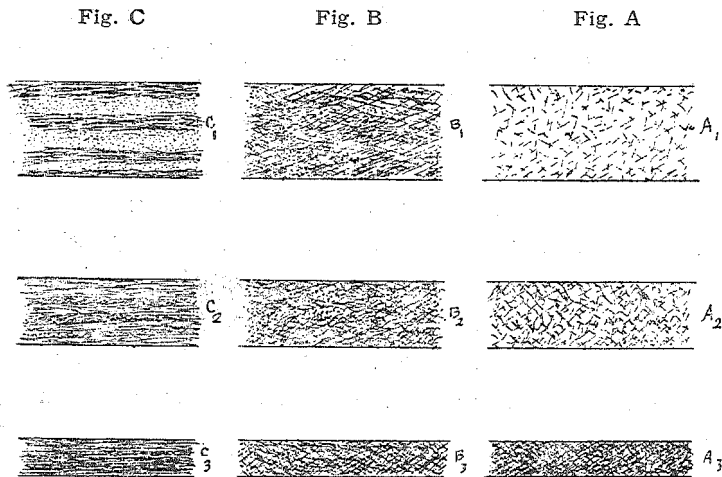


Fig. 5.

## Remarks:

In these figures, the line-like parts show the state of the silk particles, the long lines show the chain-like state caused by the high polymerization of the molecules, the short lines show the low polymerization state, the blank parts show the inter-micellar spaces, the wide blank parts show the spaces of the chain-bundles, and the scattered points in the blank show the parts of the amorphous substance.

## Explanation of the figures

## Fig. C

(Maximum stretching  
Div. 35×)

(1) Immediately after stretching.

Silk molecules are arranged in rather complete parallel, the chains assume bundle forms, water gathers in the outside of chain-bundles, the space in this part is widened.

## Fig. B

(10× stretching Div.)

Silk molecules are arranged obliquely.

The spaces are uniformly distributed but do not assume the appearance of a groove.

The spaces as a whole are large. The chain-bundles are scarcely formed.

## Fig. A

(Non-stretching Div.)

This shows the state of the particles of silk immediately after being extracted. They have a low degree of polymerization and disperse in water, taking their free directions.

They do not assume the appearance of a fiber.



<p>(2) After being left for 15 minutes.</p> <p>Moisture is evaporated and the space between the chain-bundle are much shrunk.</p> <p>The distance between the chains is only a little shrunk because it is already close at first.</p>	<p>The distance between the chains is gradually narrowed, and the chains somewhat slacken.</p>	<p>Silk molecules do not form chains and gradually shrink their spaces by the evaporation of moisture.</p>
<p>(3) Complete drying state.</p> <p>The moisture among the chain-bundles is completely evaporated and the distance between the bundles is shrunk and the chain-bundles become connected by the amorphous substance.</p> <p>The orientation as a whole becomes closest.</p> <p>The splitting quality is high.</p>	<p>Complete shrinkage takes place but the space between the chains is wider than in the case of the maximum division, i.e. the percentage of shrinkage is small.</p> <p>Spaces of molecules are rather uniform.</p> <p>The splitting quality is low.</p>	<p>Silk molecules shrink completely and are small.</p> <p>The spaces are rather large than in the case of the stretching divisions (C, B).</p> <p>There is no splitting of the molecules because they do not assume the form of the fibers.</p>

The writer already reported that in real spinning in cocooning, stretching takes place about the coagulation increasing point. (C.J.P.)

This idea justifies that the various inter-micellar spaces small and large, are formed in the silk fibers. Accordingly, let the writer examine if such spaces exist in the silk fibers. In measuring specific gravity of a fiber, it has been known that if steeping reagents of different lengths of molecules are used, different specific gravities are obtained. Dr. Matsunaga<sup>(20)</sup> measured the specific gravities of the silk fibers in various normal alcohols, and reported the following results: (Table 30)

Table 30. Relation between S.G. of Silk Fibroin and Number of Carbon Atom in Normal Alcohol Series.

	Methyl al.	Ethyl al.	Propyl al.	Butyl al.	Amyl al.	Fluid paraffin
No. of C. atom	1	2	3	4	5	10~15
Molecular length (Å)	4.7	5.7	6.9	7.9	9.0	15~20
S.G.	1.483	1.445	1.382	1.380	1.382	1.363

Remarks: S.G. where water was used: 1.426

S.G. obtained from cross-sectional area: 1.149

It has been reported by G. W. Stewart and R.M. Marrow that an average length of 1.3 Å. increases every time one atom of carbon is added.

C. H. Sogani and Stewart used a normal fluid paraffin in this measurement and could know the following relation between the number of carbons and the length of a molecule:

$$L = \frac{\text{molecular weight} \times \text{mass of a hydrogen atom}}{(4.64 \times 10^{-8})^2 \times \text{specific gravity}}$$

here,  $L$  = the length of a molecule

The value obtained from this equation was led, by plotting, to the following:

$$L = 1.24n + 1.70$$

here,  $n$  = the number of carbons

1.24 = the space between adjacent carbon atoms

From the above table, it can be observed that alcohol having short molecular length penetrates into even the small spaces and alcohol having long molecular length penetrates into only the large spaces, and therefore the various specific gravities can be got if the fiber are measured by the use of these solutions. The above table is diagrammatized as follows: (Fig. 6)

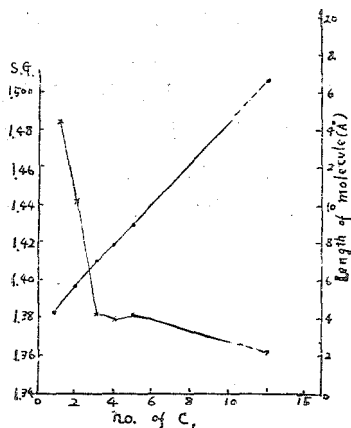


Fig. 6.

From this diagram, it can be seen that the silk fiber has two kinds of the spaces, i.e. small ones into which small particles of propyl alcohol or alcohol of less than 3 carbons penetrates and large ones into which alcohol, from amyl alcohol up to fluid paraffin, penetrates.

The former is now called the primary space, the latter the secondary space. The difference between the specific gravity

The former is now called the primary space, the latter the secondary space. The difference between the specific gravity

obtained by the use of fluid paraffin and the one obtained from cross-sectional area of silk suggests that there are the large spaces into which even paraffin penetrates. It has been thus clarified that large and small spaces are formed in the silk fiber as presumed by the writer.

Now these spaces are calculated from Tables 28, 29 and 30 as follows:

$D$ = Real S.G.	Remarks: The swelling in the
$A_1$ = Real area	direction of the fiber axis was
$G_A$ = Area S.G.	assumed to be constant.
$D_1$ = S.G. when an optional solution	
was used.	
$A_2$ = Area of the fiber when an	
optional solution was used	
$A_c$ = Cross-sectional area	
$S$ = Inter-miceller spaces occupying	
the cross section	

$$A_1 = A_c \times \frac{G_A}{D} \dots\dots (1) \quad S = A_c - A_c \times \frac{G_A}{D} = A_c - A_1 \dots\dots (2)$$

If  $D=1.483$ ,  $G_A=1.149$  and  $A_c=0.114 \text{ mm}^2$ ,

then  $A_1 = 0.114 \text{ mm}^2 \times \frac{1.149}{1.483} = 0.0883 \text{ mm}^2$

$$S = 0.114 \text{ mm}^2 - 0.0883 \text{ mm}^2 = 0.0257 \text{ mm}^2$$

Namely, in the case of 5 times stretching, the inter-miceller space becomes 22.53%. (S.G. 1.3797) Now, taking  $D_1$  to be 1.382 and 1.363, the spaces into which  $A_2$ , propyl alcohol and fluid paraffin can penetrate are obtained as follows:

Table 31.

Alcohol	S.G.	Part of fiber	Part of space of fiber
Methy alcohol	1.483	0.08831 mm <sup>2</sup>	0.0257 mm <sup>2</sup>
Propyl alcohol	1.382	0.09403	0.01997
Fluid paraffin	1.363	0.09534	0.01866
Cross sectional area	1.149	0.114	0

Remarks:

It was assumed that the fiber does not cause swelling if it is steeped in the solution.

These spaces are the parts where each solution can penetrate into the cross-sectional area of  $0.114 \text{ mm}^2$ . This is now diagrammatized as follows: (Fig. 7)

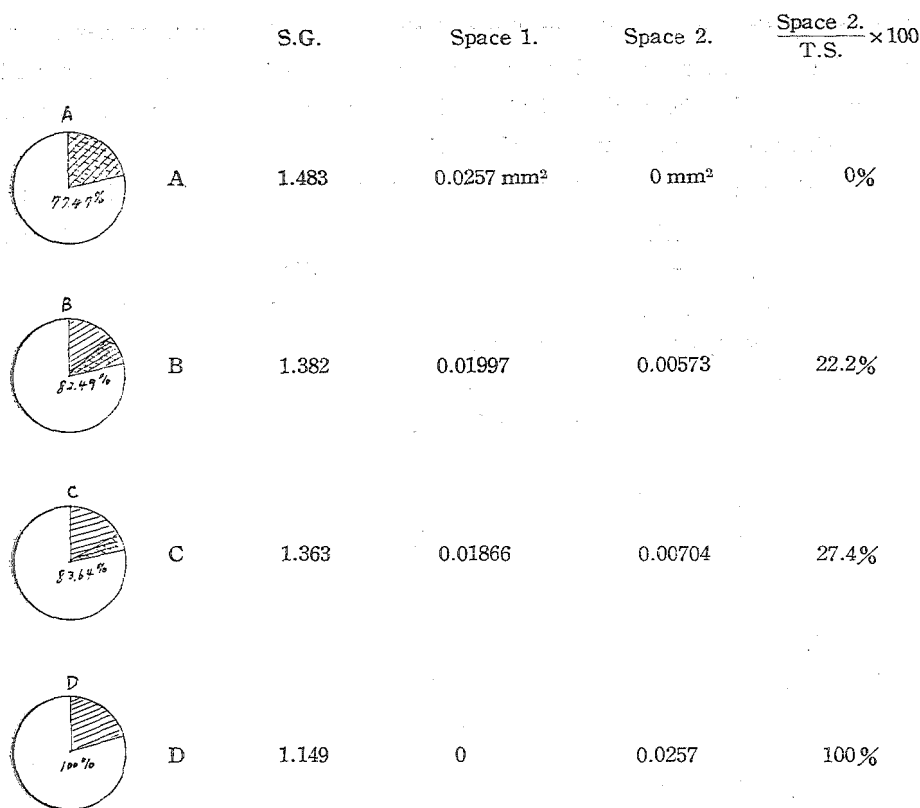


Fig. 7.

## Remarks:

The blank in the circle shows the fiber substance, and the diagonal part shows the space. The dotted diagonal part shows the spaces into which solutions having these S.G. can penetrate.

Space 1. ....Area of inter-miceller space infiltrate of optional solution.

Space 2. ....Area of inter-miceller space does not infiltrate of optional solution.

T.S. ....Total inter-miceller space.

Blank part in the Fig. 7, that is indicate true area of fiber, and part of oblique line indicate inter-miceller space.

Part of oblique dotted line indicate space infiltrate on alcohol solution of each specific gravity.

The specific gravity is 1.363 in the case of paraffine and also is 1.149 in the case of the cross-sectional area. It is therefore assumed that the silk

fiber has spaces of over 15~20 Å, into which the paraffin can also penetrate to some extent. Frey-wyssling let 0.5% auric chloride (AuCl<sub>3</sub>) penetrate into the fiber and then reduced it. From the sizes of these particles, he measured the space of 50 Å. Thus it can be assumed that there exist various sizes of the inter-micellar spaces i.e. from 3 Å, the smallest, to 50 Å, the largest, in the silk fiber. In case the fiber is constituted under the condition of different multiples of stretching as described before and also in the case of the division of lower multiples of stretching where the orientation of micells is at random, there is no clear distinction between the primary and the secondary spaces, and the differences in sizes of the spaces are little, but the spaces are generally large and the specific gravity is small. If the multiple of stretching is increased and so the micell orientation is improved, the distances between the chains, and between the chain-bundles are both narrowed, and the constitution as a whole becomes fine and close, and therefore the specific gravity becomes larger. The silk substance obtained from the experiment which is indicated in the Tables 28 and 29 was degummed and then its specific gravity was measured, with the following results:

Table 32. Relation between S.G. of Fibroin and Degree of Stretching.

Degree of stretching	Non-treated		Treated	
	boil-off loss	S.G.	boil-off loss	S.G.
Non-stretching	44.6%	1.3765	45.4%	1.3636
5 times	37.7	1.3923	43.4	1.3797
10 times	41.2	1.4055	46.1	1.3911
15 times	38.1	1.4210	44.2	1.4110
35 times (max. stretch)	40.1	1.4722	38.7	1.4757

As seen from these results, if the stretching multiple increases, the specific gravity increases, this relation being in a straight line i.e. the fiber becomes close by means of stretching. Regarding the relation between the stretching multiple and the swelling degree, we can know that the swelling degree becomes higher in accordance with the increase of the stretching multiple and so the relation is in a straight line, about which the writer will report separately. Concerning the specific gravities of the cocoon fibers, they are perceived to be large about the middle layer and to be small about the outer and inner layers as reported by the writer<sup>(21)</sup> and Prof. Kubota.<sup>(22)</sup> And the swelling degree is perceived to be large at the middle layer having a large specific gravity and to be small at the outer and inner layers. The same

relation is quite true of the artificially stretched silk gut, which fact is sufficient to affirm the writer's opinion.

The fact that much lousiness generates at the middle layer having a large specific gravity shows that the middle layer has the good orientation of micells. It can be also thought that the origination of the branched fibers at the middle layer has something to do with the origination of the lousiness. This is because the branched fiber has higher splitting quality than the main fiber. Explanation is possible regarding the dyeing properties of the main fibers and of the fine fibers produced by such splitting i.e. the absorption of dyestuffs is done chiefly at the amorphous parts or inter-miceller spaces and therefore the main fiber containing many amorphous parts is deeply coloured, while the split fine fiber is light-coloured because it has few amorphous parts and is close in constitution and is good in high-transmission. This explanation also makes clear the factors promoting the unevenness in the colouring of the split fibers and the main fibers.

So the writer insists. Next, let him consider the cohesion acting among silk molecules in time of swelling. Such free ions as groups  $-\text{CO}-\text{NH}$ ,  $-\text{NH}_3^+$ , and  $-\text{COO}^-$ , in a silk molecule, which form the center of the electric field, strongly attract and closely arrange dipoles of water adjacent to them. The farther the water molecules are from the field, the more disorderly they are arranged. Further when they are far more remote from the field, the moisture comes to be in the scattering state and ultimately becomes so-called "free water". With the increase of the swelling degrees, the moisture in the silk fiber assumes such a condition. Considering the evaporation of the moisture shown in the previous table (Table 28, 29), the evaporation of the free water takes place early, and according to the condition of air, takes place in order of moving-water and bound-water, and at last stops.

If the silk fiber is swelled in water of about 7.0 pH, the solidarity or cohesion of the chains and chain-bundles is loosened and the distance between the chains is widened and at the same time there occurs the penetration of solution. Accordingly the moisture penetrates the primary and the secondary spaces. Although each space is widened, the part where the micells are interlaced has a low percentage of enlargement, while the part where the orientation of micells is good has a high percentage of enlargement as the chain-bundles are widened. These micells are connected one another by the secondary force of Van der Waal's force and the cohesion increases in inverse ratio to the cube of the distance. Therefore there are great differences in the cohesion of the micells widened by swelling, because of the differences in the

percentage of widening. Now, let an average swelling degree of the silk fiber in water be 35% (if the swelling took place only at the amorphous parts of the chain-bundles and inter-micellar spaces, it would be carried out by the solvation of the amorphous parts or micell surface.) and let the ratio of the crystalline part to the amorphous part be about<sup>(23)(24)(25)</sup> 8 to 2 in the case of natural fibers such as silk fibers, and the variation in the molecular cohesion between in the dry state and in the swollen state is obtained as follows:

$$0.8 + (1+X)0.2 = 1.35 \quad X = 1.75$$

Here,  $X$ .....Percentage of swelling  
1.35... ..Average swelling degree of silk

Let the width of a fiber be 1, and the swelling degree is 2.75. The cohesion varied by the swelling is as follows:

$$F = F_0 \times \frac{1}{(2.75)^2} = \frac{F_0}{20.8}$$

Here,  $F$  = Cohesion after swelling  
 $F_0$  = Cohesion when dry.

Now, the variation between the water-swelling of silk and  $F$  is obtained as follows:

	Swelling of cocoon layer	Swelling percentage ( $X+1$ )	$F$
Outer cocoon-layer	19.0%	194%	$\frac{F_0}{7.41}$
Middle cocoon-layer	33.5	267	$\frac{F_0}{19.0}$
Inner cocoon-layer	24.0	220	$\frac{F_0}{10.6}$

From the Table 30, the primary space is expressed in  $4.3-6.9=5.6 \text{ \AA}$  and the secondary space, in  $7.9-20.0=14 \text{ \AA}$  respectively, and then the difference in the cohesion between the two spaces is  $\frac{F_0}{(5.6)^3} : \frac{F_0}{(14)^3} = \frac{F_0}{169.6} : \frac{F_0}{2744.0}$  in which the difference is clearly seen.

When the swelling as indicated in the above table is performed, it is assumed that the primary spaces are found numerous in the outer and inner cocoon-layers and that the secondary spaces are found numerous in the middle cocoon-layer, and the difference in the cohesion between the two after swelling becomes larger and thus the possibility of splitting at the middle cocoon-layer still increases.

For example, the ratio of the swelling degree between the outer and middle cocoon-layers is:

$$\frac{F_0}{159.6 \times 19} : \frac{F_0}{2744 \times 33} = \frac{F_0}{3222} : \frac{F_0}{90552}$$

and the ratio of the outer layer to the middle layer is 1 to  $\frac{1}{28}$  which means a great difference in the cohesion between the two. If the amorphous substance in these spaces dissolves, splitting takes place perhaps irreversibly. And in case such spaces are formed in parallel over a length, splitting takes place continuously. It is by the above consideration that the splitting of the silk fiber and the formation of the lousiness caused by the splitting can be explained theoretically.

## VII. Summary

The writer could learn the following facts from these studies.

1. The writer pointed out the splitting quality of the silk fiber as the cause of the formation of the lousiness.
2. The number of the lousiness-spots is varied by different degrees of degumming. The degrees of degumming are varied by the concentration of the treatment solution and by the time and temperature of treatment, the temperature, among others, being the factor having a strong effect on the origination of the lousiness.
3. As the treatment temperature is elevated, the number of the lousiness-spots increases, and the time in which the lousiness-spots become most numerous shortens; for instance, it was 60 hrs. for 80°C, 20 hrs. for 100°C, 3 hrs. for 120°C, and 1 hr. for 130°C. The numbers of the lousiness-spots corresponding to these were 52, 64, 71, and 85 respectively. If further treatment is done, the lousiness gradually decreases and finally vanishes.
4. The origination of the lousiness caused by the degumming process differs according to the silkworm kinds. The kinds the fibers of which are hard to split have very low variation, while the kinds the fibers of which are easy to split have very high variation.
5. In case re-degumming is done after removal of the lousiness which is generated by the regular degumming, the fibers of the kinds of easy-splitting generate lousiness again.
6. As the maturing degree of the silkworm is enhanced, the number of the lousiness-spots increases. This is due to the enhancement of the maturing degree of the liquid silk and also to the fast spinning velocity.



7. The kinds having such construction of the spinneret that makes silk strongly pressed in passing through it, generates much lousiness.

For example, the cocoon fiber of the silkworm kinds having much liquid silk in proportion to the dimension of the spinneret or having a partly chitin board in the lower part of the spinneret (ventral sclerotized bar) generates much lousiness. This is due to the strong pressure which silk receives in its passing through the spinneret and also to the better orientation of silk micells by rubbing friction.

8. The cohesive power between the molecules or micells of silk weakens as the inter-micellar space widens. Therefore if the cohesive power between the micells is weakened by swelling, the splitting of the fiber takes place.

9. The splitting quality of the cocoon fiber is generally strong at the middle layer. This is because the spinning velocity of the middle layer is high and the stretching is done up to about the increasing point (C.I.P.) and the silk is strongly pressed in the orifice because of the thickness of the fiber. The fiber of this part is close in constitution. The percentage of swelling in width is always high. Therefore the micell orientation is good and the splitting quality is strong.

10. Regarding the dye absorptiveness of the main fibers and the lousiness-spots, the main fibers have higher affinity for acid or basic dyestuffs than the lousiness-spots, and the lousiness-spots have higher affinity for direct dyestuffs. In both cases, however, the lousiness-spots appear to be of light hue. This cannot be explained only by the difference in the degree of colouration between the two as hitherto described by other researchers. The writer should like to point out, as one of the causes, that difference in the amount of transmitted light which comes from the extreme difference in thickness of the dyed materials.

11. As the cause of the formation of the branched fibers, which are one of causes of the lousiness, the writer pointed out separate fibroin and plait-like formations on the surface of the liquid silk in the silk-gland. He assumed that the formation of the separate fibroin and plait-like parts is due to the difference in the maturity coming from the different stages of the secreting fibroin substance and to the pressure from the posterior division of the gland.

12. The branched fiber and the plait-like part of the outside part of the main fiber are high in width-swelling degree as compared with the central part of the main fiber. This is responsible for the fact that the splitting quality of the silk formed from the liquid silk of this part is high.

13. When the silk fiber is formed, the micell orientation of the fiber is

either uniformly performed or ununiformly performed. In the former case all of the silk has the splitting quality, while in the latter case the silk has partially the splitting quality.

14. There is a limit in the thickness of the separate fine fiber to start a spiraling motion. In the materials used in this experiment, it was an average of about  $1.0\ \mu$ , with the thickest of  $3.0\ \mu$ . The thickness differs according to the quality and temperature of the steeping solution.

15. The fact that the width of a fiber to start the spiraling motion is within the limit of about  $1\ \mu$  is very interesting, for it seems to have some connection with the Brown's motion which takes place in small particles.

16. The writer thinks that the spiraling motion of the separate fiber is based on the partial difference in the swelling of the fiber from a constructional point of view, i.e. it comes from the partial difference in the micell orientation.

17. The origin of the splitting of the silk fiber is as follows: as the constitution of the silk fiber is different from part to part, the difference in the swelling degree occurs in the silk when it is swollen in the processes of degumming and dyeing. And the cohesive force between the micells differs from part to part. Therefore, splitting happens at the parts where the cohesive force is extremely low against the same outside force and also where the micells are orientated in parallel.

Furthermore, these parts form masses by the spiraling action, which is the secondary cause of the lousiness. As for the cocoon fiber, numerous factors for decreasing such cohesive power gather in the middle cocoon layer, where splitting happens most remarkably, thus forming the lousiness.

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## IX. Explanation of Plates

- Fig. 1. A shape of lousiness, yield by split of fibroin.....Split form.
- Fig. 2. A shape of lousiness, yield by aggregation of split fibroin.....Aggregated form.
- Fig. 3. To shows disintegration process of fibroin.
- A. To shows partialy disintegration of fibroin.
  - B. To shows disintegration in whole parts of fibroin. In somewhat length, lousiness is not make up in such disintegration. (split)
  - C. Split is stronger than B, and split fiber is finer than B, but yet do not maked lousiness.
  - D. Diameter of split fiber is becomes  $1\sim 3\mu$ , in such size of split fiber, it become to lousiness.
  - E. Fibroin disintegrate to almost all parts of fibroin and formation of lousiness is very exceedingly.
  - F. Split fiber is break out in any point of fibroin and to turn down to aggregation, and lousiness is formed from this split fiber.
- Fig. 4. To shows the cross-section of split fiber is formed from fibroin.
- A. To shows biginning disintegration of fibroin in partially.....A arrow mark part.
  - B. This figure is the cross-section in center of Fig. 3. E, it is still remain non split part of fibroin.
  - C. This figure is the cross-section in center of Fig. 3 F, it is split out almost perfect disintegration.
- Fig. 5. Shape of spinneret of silkworm. ....*Bombix Mori*.
- Form A. 3 minsan.
  - Form B. (1) Chinese 17, lousiness severe.
  - Form B. (2) Europe 19×Chinese 17.
- Fig. 6. The cross-section of dry and swelling fiber.
- A. The cross-section of dry samples of cocoon fiber.
  - B. The cross-section of swell up sample for 80% of HCOOH.
- Fig. 7. Sepalate fine fiber of fibroin by difference of degumming process.
- A (+) B (++) C (###) of figure indicate from the slightly separate fiber to severe one in each sample.
- Fig. 8. To indicate cross-section of cocoon fiber by difference of kinds of silkworm, swell up for 80% HCOOH.
- A. ....Seihaku.
  - B. ....3 minsan.
  - C. ....Chinese 17 lousiness few.

- D. ....Kuwako×Kasan.      E. ....Europe 16.      F. ....Chinese 4.  
 G. ....Europe 18.      H. ....Chinese 16.  
 I. ....Chinese 17, lousiness severe.      J. ....Japanese 112.  
 K. ....Tegususan.

Fig. 9. Branched fiber in cocoon layer and split fiber in the degummed silk.

- A. ....Cocoon fiber of 3 minsan. A' .....degummed silk of A.  
 B. ....Cocoon fiber of Chinese 17, lousiness severe.  
 B' .....degummed silk of B.

Fig. 10. The gathers of coagulations of liquid silk in the silk-gland.

- A. A'.....Surface of fibroin substance, arrow mark show the direction of translation of silk substance.

Fig. 11. To shows the longitudinal section (B) and the cross-section on the gather parts of fibroin substance in the silk-gland.

- Fig. 12. A .....Photo. of surface on the gather parts of fibroin in the silk-gland.  
 B .....Photo. of longitudinal section of controled silk-gland.

Fig. 13. To shows that branched fine fibroin on the surface of coagulations of liquid silk in the silk-gland. (After degummed) f.....main fibroin. b. q..... branched fiber.

Fig. 14. To shows that separate fine fiber on surface of fibroin on coagulations of liquid silk in the silk-gland. (After degummed)

- j. ....Normal shape of fibroin.  
 K. ....Deformed shape of fibroin.  
 D' .....Photo. of branched fiber on the fibroin in the silk-gland.  
 K' .....Photo. of separate fine fiber on the fibroin in the silk-gland.  
 Sr.....Sericine. SG.....Cell wall.

Fig. 15. To indicate the modell of destroyed fibrils, which is latent in the coagulate fibroin in the case of stretching at coagulation increasing point. (C.I.P.)

Fig. 16. A modell of breaking part of fibroin caused by such as Fig. 15. S.....split part.

Fig. 17. To indicate on the orientation of fibrils in the degummed fibroin to yield by difference for degree of stretching.

- A. ....Ununiformly orientation of fibrils, 5 multiple in stretching.  
 B. ....Uniformly orientation of fibrils, 8 multiple in stretching.  
 C. ....The almost perfectly orientation of fibrils (15 multiple in this treatment)

To shows the partialy splitting as fibrils is ununiformly orientation.

- D. ....To indicate perfectly coagulate stretching of liquid silk, the orientation of fibrils is almost uniformly, and split of fibroin is somewhat severe.  
 E. ....To indicate perfectly coagulate stretching (C. I. P.) of liquid silk,

the orientation of fibrils is almost uniformly, and the split of fibroin to yield and it is brought about on whole parts of fibroin.

- Fig. 18. The split of fibroin in the case of swelling up with 1.5% NaOH.
- Fig. 19. The deformation of split fiber.....wave like shape.
- Fig. 20. Form N and O is the deformation of split fiber..... Spirally like shape.
- Fig. 21. A kind of very fine split fiber caused by disturbed of water. (Solution)
- Fig. 22. A model of split fiber yield by swelling action.

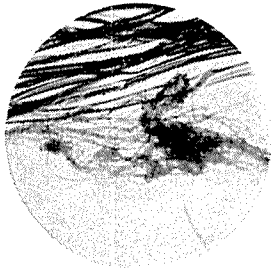


Fig. 1

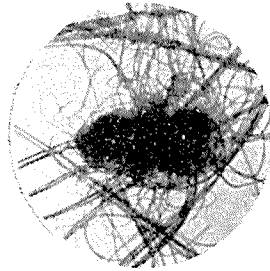


Fig. 2

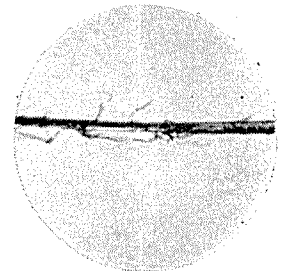


Fig. 3 A

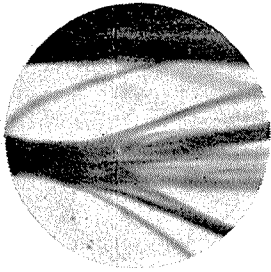


Fig. 3 B

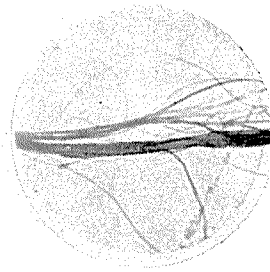


Fig. 3 C

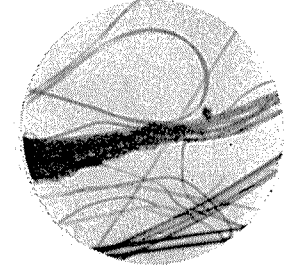


Fig. 3 D

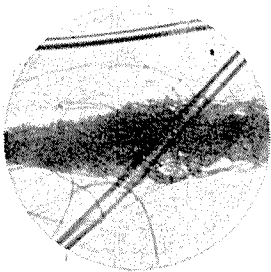


Fig. 3 E

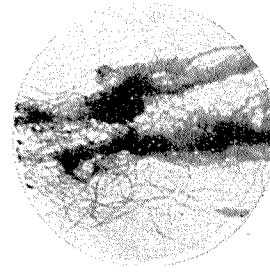


Fig. 3 F

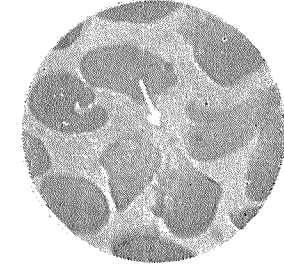


Fig. 4 A

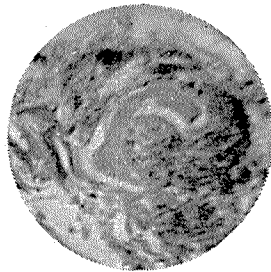


Fig. 4 B

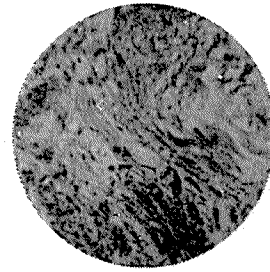


Fig. 4 C

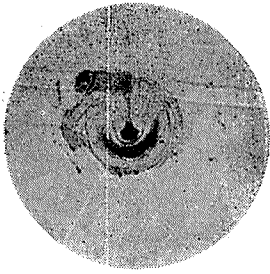


Fig. 5 Type A



Fig. 5 Type B (1)



Fig. 5 Type B (2)



Fig. 6 A



Fig. 6 B

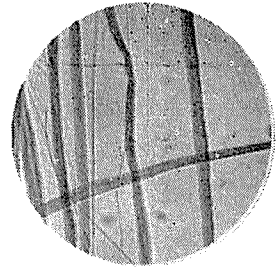


Fig. 7 A (+)

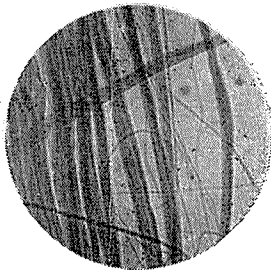


Fig. 7 B (++)



Fig. 7 C (+++)

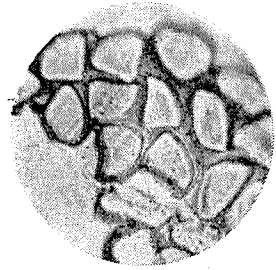


Fig. 8 A



Fig. 8 B



Fig. 8 C

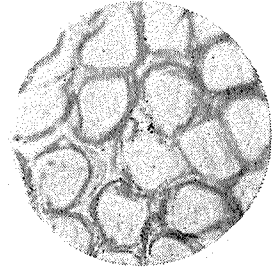


Fig. 8 D



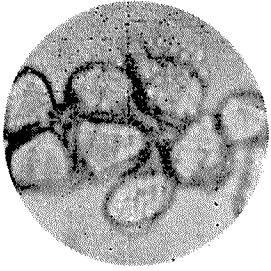


Fig. 8 E



Fig. 8 F

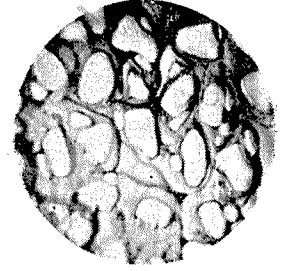


Fig. 8 G

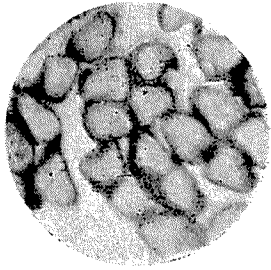


Fig. 8 H



Fig. 8 I

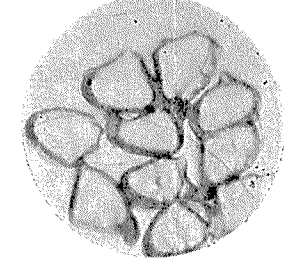


Fig. 8 J



Fig. 8 K



Fig. 9 A

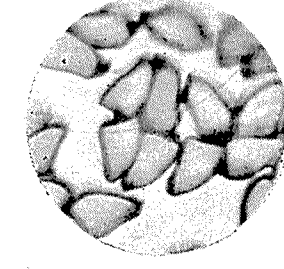


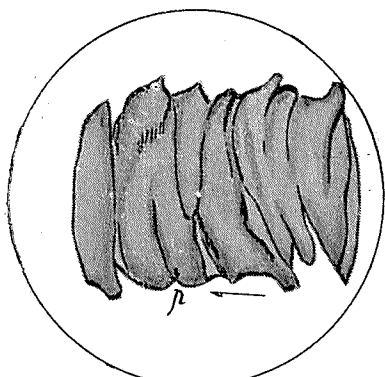
Fig. 9 B



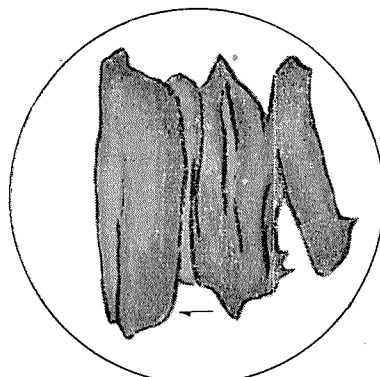
Fig. 9 A'



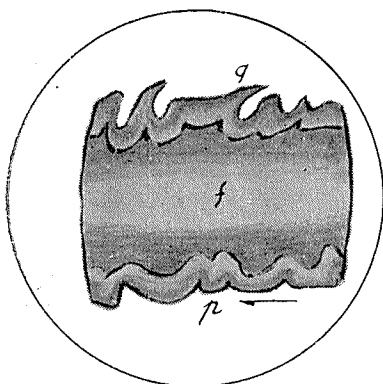
Fig. 9 B'



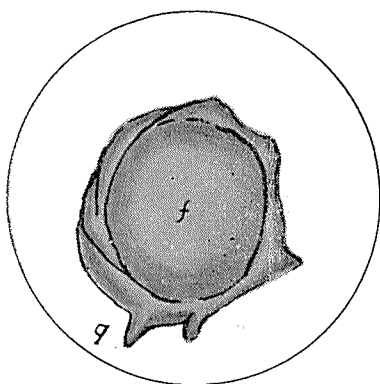
*A*  
Fig. 10 A



*A'*  
Fig. 10 A'



*B*  
Fig. 11 B



*C*  
Fig. 11 C

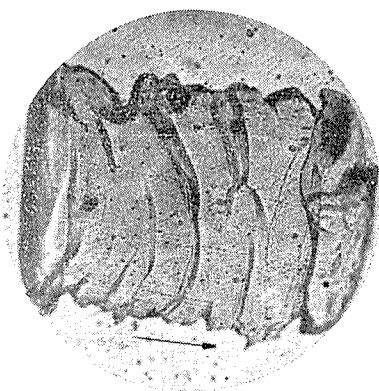


Fig. 12 A

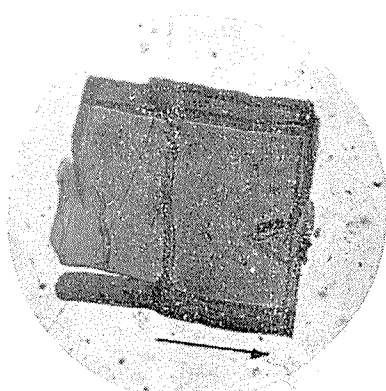
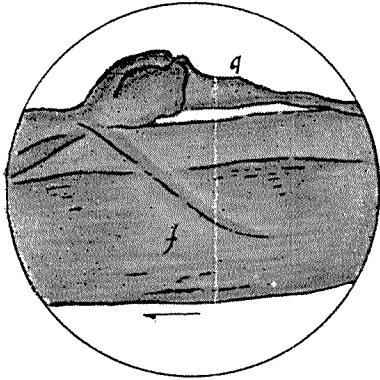
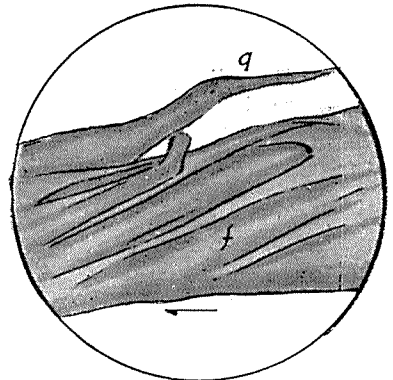


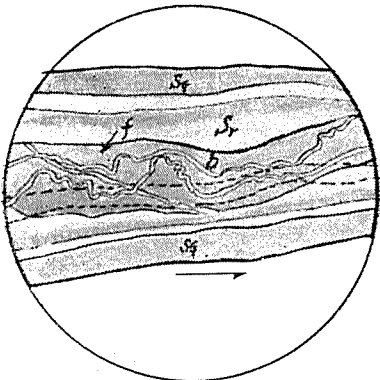
Fig. 12 B



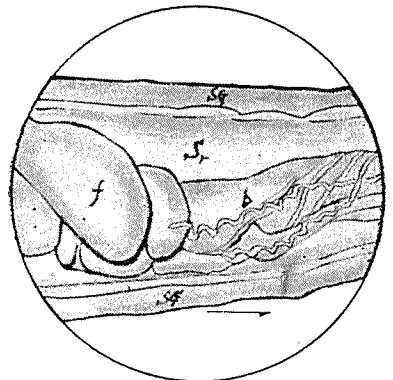
*D*  
Fig. 13 D



*E*  
Fig. 13 E



*J*  
Fig. 14 J



*K*  
Fig. 14 K

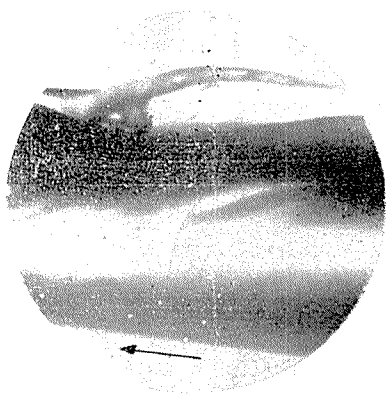


Fig. 14 D'

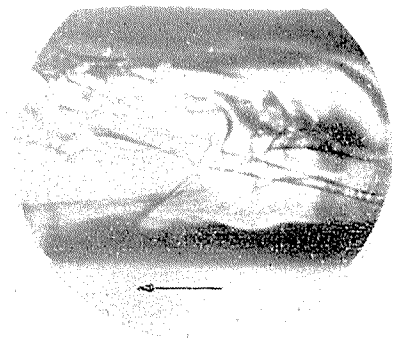
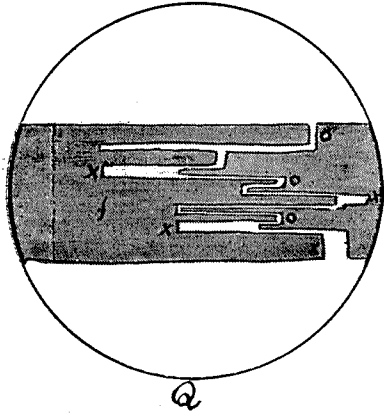
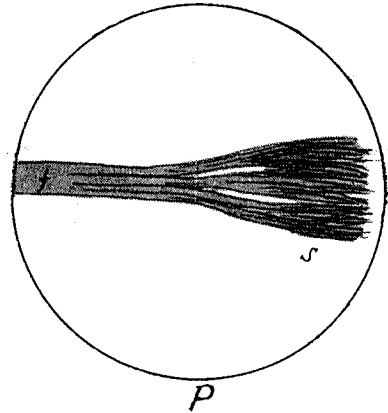


Fig. 14 K'



*Q*  
Fig. 15 Q



*p*  
Fig. 16 P

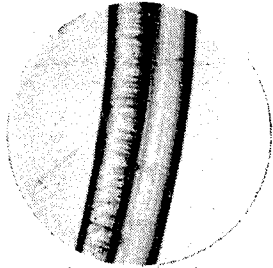


Fig. 17 A

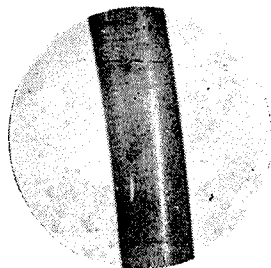


Fig. 17 B

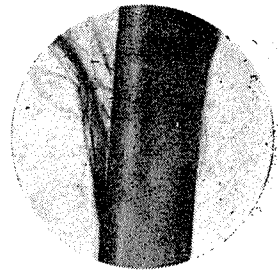


Fig. 17 C

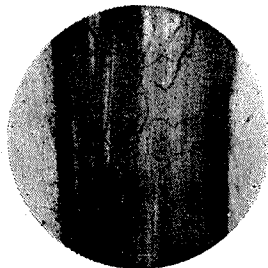


Fig. 17 D

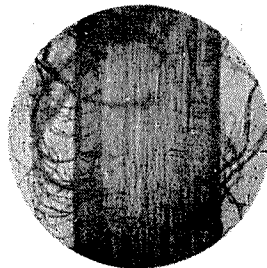
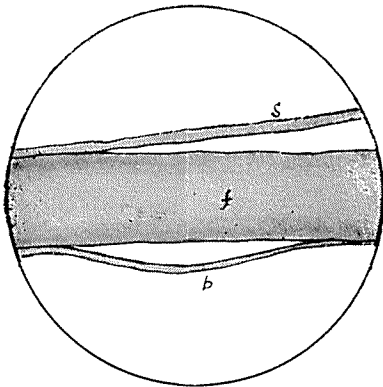
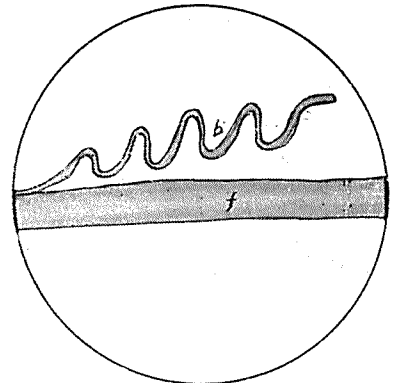


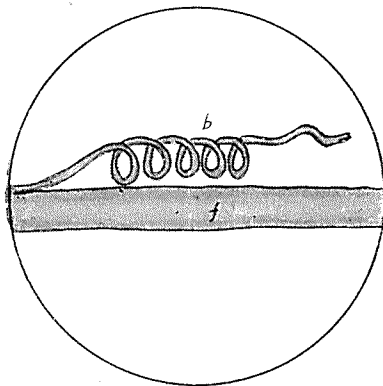
Fig. 17 E



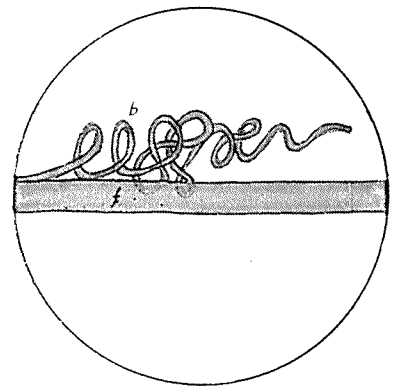
*L*  
Fig. 18 L



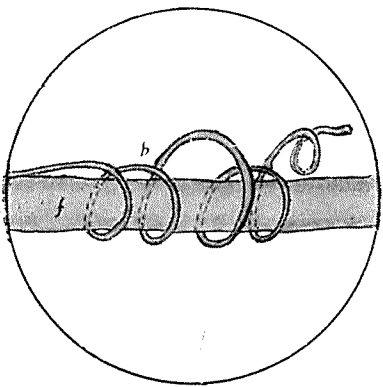
*M*  
Fig. 19 M



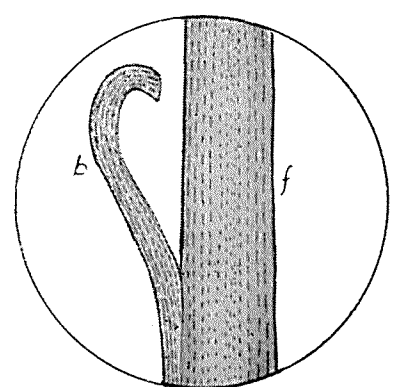
*N*  
Fig. 20 N



*O*  
Fig. 20 O



*R*  
Fig. 21 R



*I*  
Fig. 22 I