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FULL PAPER

NON-DESTRUCTIVE EVALUATION OF HISTORIC TEXTILES BY COMPRESSION MEASUREMENT USING THE "KAWABATA EVALUATION SYSTEM (KES)"

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To propose a new non-destructive assessment procedure for historic *Suga*-yarns using the "Kawabata Evaluation System (KES)", compression resilience (RC) was compared with tensile breaking strain, which is traditionally used to measure mechanical properties of yarns. Modern raw silk yarns dyed with tannic acid and iron mordant were degraded artificially and used as a model of historic *Suga*-yarns. The tensile breaking strain of model yarns decreased over a 70-days degradation period, so did the RC.

The mechanical properties of model yarns were categorized into "Strong", "Weakened" and "Poor" in the order of decreasing mechanical properties. Since the SEM observations showed no damage in the morphology of yarn surfaces after compression by the KES compression tester, it was found that the compression test is a better and non-destructive evaluation method for yarns than tensile testing. The historic *Suga*-yarns exhibited approximately 20% - 40% of RC, and less than 7% of the tensile breaking strain. Their physical condition was classified as "Weakened" and "Poor". This suggests that mechanical condition evaluated on the basis of model yarns can be applicable to historic *Suga*-yarns.

1 Introduction

In the conservation of textile fibres, it is common to evaluate the condition of an object by assessing the physical integrity of its structure and its chemical stability. From a variety of analytical techniques available for historic textiles, as is common with all cultural heritage items, non-destructive testing and minimal sampling methods are the preferred techniques or the prerequisite specified by the owner.

For many years, the authors have been involved in research on determination and reduction of damage to ceremonial Japanese doll's hair. Japanese ceremonial dolls of the late Edo period, called "*Hina* dolls", were a symbolic item used during the Girl's Day festival ("*Hina matsuri*") at the time. The *Hina* doll's hairs were usually made of "*Suga*-yarn", raw silk yarns dyed black. It is known that yarn degradation is caused by a

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combination of acid-catalysed hydrolysis and iron-catalysed oxidation.[1-3] Research using European metal-weighted silks, weighted with tin phosphate/silicate in the late 19th and early 20th centuries, showed similar problems.[4,5]

Methods for evaluation of mechanical properties generally used in the field of textile conservation science are typically tensile breaking strength, tensile breaking strain and/or tensile initial modulus.[6-8] On the basis of previous research on conservation treatment of *Suga*-yarn, tensile properties of model yarns were investigated.[9,10] However, the problem with this test is that a considerable amount of sample yarn is generally needed. In addition, at present, heavily degraded surfaces are often observed on *Suga*-yarns and it is difficult to measure tensile properties on such yarns.

In 1970, a series of instruments to measure mechanical and surface properties of textiles was devised and data was obtained that replaced subjective assessment of fabrics with objective, instrument-based, consistent and reproducible results. The Kawabata Evaluation System (KES) of textiles consists of instruments for testing, including tensile, bending, shear, compression, and friction testing.[11,12] The KES system has been widely used, not only for fabrics, but also in the fields of cosmetic, food, paper and automotive industry. The compression tester, the KES-G5S, can measure the compression force-displacement using various types of “contactors” depending on the shape of the tested object. It can measure compression energy, resilience, and rigidity with high accuracy. When measuring yarn filaments with KES-G5S, yarn pieces of approximately 2-3 mm length are needed. In addition, three-dimensional objects, such as cosmetic sponge, powder puff and *futon* padding, have been measured directly without sampling by the KES compression tester.[13-15] In the case of a textile object, it can be placed on a wide open stage and compression properties are detected by the compression contactor moving up and down and gently touching the sample. Provided that there is a relationship between the currently available tensile testing and the compression values measured for historical textiles, a non-destructive evaluation method using small-scale samples may be made available.

In practical preservation and conservation work with *Hina* doll’s hair, understanding of the mechanical properties is one of the most significant factors for long-term preservation. It is expected that the assessment of compression properties will help conservators understand the mechanical properties of the materials in question.

In the present study, modern raw silk yarns were dyed with tannic acid and iron mordant to model historic *Suga*-yarn. During an accelerated degradation process at high humidity and high temperature, the effects of degradation on mechanical properties of model yarns were investigated using the KES tensile and compression testers. The mechanical state of each yarn was estimated by measuring the tensile strength, the breaking strain, the compression displacement and the compression resilience. The influence of compression pressure on yarn surface morphology was evaluated by scanning electron microscope (SEM).

Based on the results, a suitable compression method was developed to find out the characteristic mechanical properties of historic *Suga*-yarns.

2 Experimental

2.1 Materials

Throughout this research, multiple-reeled *Iyo* raw silk (2.61 tex) produced in Ehime Prefecture was used. It is *Akebono* of the bombyx cultivar. The Japanese word *Iyo* comes from the *Iyo* Province in Japan in the area of what if today Ehime Prefecture. The raw silk was rinsed with de-ionized water at ambient temperature, dried naturally, and used for the experiments. Tannic acid and ferrous sulfate heptahydrate were purchased from Nakarai Tesque, Ltd. As for original historic *Suga*-yarns, four *Kyoho Hina* doll’s hairs were used. The samples were abbreviated as S1, S2, S3 and S4. Figure 1 shows one of the investigated *Hina* dolls (S4) made in the late Edo period (private collection). The cross-section picture of S4 is shown in Figure 2. It has been estimated that raw silk yarn of around 21 denier, usually made of 7 or 8 cocoons, was used for *Hina* doll hair.

2.2 Methods

The raw silk yarns used as artificially degraded samples were dyed with tannic acid aqueous solution at 20 °C for 1 h, then treated with an aqueous solution of ferrous sulfate at 20 °C for 30 min after rinsing them with de-ionized water to wash off excess tannic acid. The concentration of tannic acid was 1 wt% and that of ferrous sulfate was 2 wt%. The solution-to-yarn

Figure1: Front view of the *Kyoho-hina*.

Figure2: Cross-section of the *Kyoho-hina* doll's hair.

ratio was 100:1 for tannic acid and 50:1 for ferrous sulfate. After rinsing the yarns in de-ionized water, they were dried in a ventilated oven at 50 °C for 30 min. The samples treated with 1% tannic acid and 2% ferrous sulfate solutions are abbreviated as "TAFe" hereafter.

Accelerated degradation was performed following the ASTM5032-97 standard. Modern silk samples were artificially aged in a desiccator filled with a solution of glycerol-water (58% w/w) maintaining relative humidity (RH) at 75% at 70 °C for 0, 28, 42, 56 and 70 days.[16,17] These conditions were chosen to investigate the effects on the mechanical properties of the model yarn both before and after the degradation process under high humidity at high temperature. Photodegradation was not of interest as *Hina* dolls are generally stored in darkness, such as a storage areas, at high humidity.

2.3 Measurements

Compression tests were carried out using a compression tester (KES-G5S, Kato Tech. Co., Ltd.). As shown in Figure 3, the yarns were placed on a glass slide. A contactor compressed the perpendicular yarn axis at a compression speed of 10 µm/s to a maximum force of 0.1 N at room conditions. The yarns were compressed with the maximum force and did not break under these conditions. The flat contactor was 5 mm wide and 0.2 mm thick. The compression area in contact with the contactor was 0.01 mm^2.

The compression contactor was placed on the yarn surface and measurement started. Mesurements were carried out at least twenty positions on each yarn. The raw silk yarn represented a bundle of fibres, rather than a single fibre. However, unlike a degummed silk yarn, raw silk yarn exhibited sericin on the surface, covering each filament and binding each other, and was not twisted. Since the raw silk yarn did not move during the compression measurement, compression mode for single fibres was used in this study.

Figure 4 shows typical pressure-displacement curves that can be used to calculate the characteristic parameters of compression displacement (δD) and compression resilience (RC). The δD values were obtained under maxium force of 0.1 N, at the distance of Q_1Q_3 as described in Figure 4. Additionally, the RC value is the percentage of recovery in yarn diameter after removal of the applied force. The RC was calculated using Equation (1).[18]

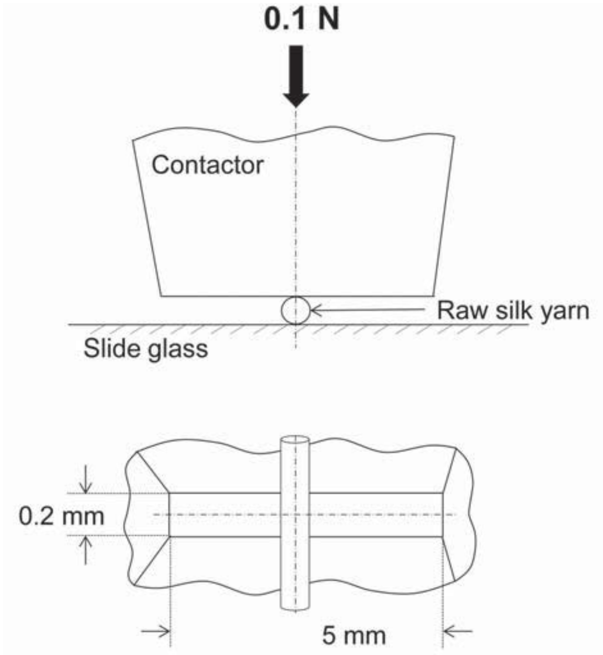

Figure 3: Diagram of the contact area for compression tester KES-G5S in the transverse direction.

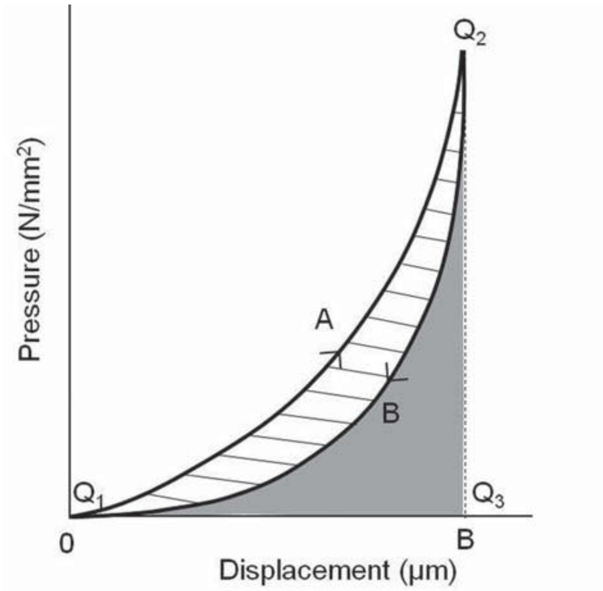

Figure 4: Typical pressure-displacement curves for the compression test. Curve A represents the compression process and B the recovery process after reaching a maximum force at Q_2. WC(Compression energy)=[area Q_1 A Q_2 Q_3]. RC=(Compression resilience)=[area Q_1 B Q_2 Q_3]/WC. δD=(Compression displacement)=Q_1 Q_3, displacement at the maximum force.

$$RC = \frac{\text{Area under load decreasing curve B (grey area)}}{\text{Area under load increasing curve A (grey and shaded area)}}$$

Equation 1

The statistical significance of differences between the δD and RC values of the degraded yarns was investigated using the t-test. In all evaluations, the difference was considered significant at the value of $P<0.05$.

The tensile test was carried out using a raw silk yarn of 10-mm length, using a KES-G1S tester (Kato Tech Co., Ltd.) at an extension rate of 0.1 mm/sec. Each time 20 TAFe yarns and 10 historic *Suga*-yarns were used for measurements of breaking strain (distortion until yarn is broken (%)) and strength (tensile force required to break the yarn (cN/dtex)). A holder for raw silk yarns was prepared following the JIS-L-1095 standard. The yarns were cemented into individual cardboard frames by using Aron Alpha *α*-cyanoacrylate adhesive (Konishi Co., Ltd). The cardboard frames had an opening window of 1 cm^2. The samples were left in the frames for a day for the cement to set. They were then gripped by the clamps and the sides of the frame were cut away leaving the yarn between the clamps before they were loaded into the tensile tester.

The TAFe surface was observed using a JSM-7001F SEM (JEOL Ltd.). All the yarns were gold-coated over a 2-cm length using ion sputtering equipment (JEOL Ltd., JFC-1100E) at 6 mA for 4 min prior to the SEM measurement.

3 Results and Discussion

Figure 5 shows typical stress-strain curves for the TAFe yarns after degradation. The strength and breaking strain results are plotted against dergadation time in Figure 6. The strength of the TAFe yarns decreased approximately 21% and 95% after 28 day and 56 day of degradation, respectively. The breaking strain of TAFe yarn decreased by 50% and 94% in the same time periods, respectively. The silk yarns degraded for longer than 56 days are easily pulverised when picked with tweezers, which was similar to the hair of *Hina* dolls from the *Edo* period.

In Figure 7, pressure-displacement curves depict compression characteristics for TAFe yarns during the 70-day degradation period. From these curves, the compression displacement δD and the compression resilience RC were calculated. The δD describes how much the yarns are compressed by the contactor. Therefore if the yarns become rigid, the compression displacement increases. As the value of RC signifies yarn recovery, the degraded yarns have lower RC than yarns before degradation. Although the values of δD and RC was calculated from the condition of yarns before breaking, δD and RC of the heavily degraded model yarns were close to the δD and RC at breaking point.

The value of compression displacement δD and compression resilience RC is plotted against degradation time for TAFe yarns in Figure 8. Although the δD increased from 16 μm to 20 μm during the period of degradation of 70 days, no statistically significant differences between the values of δD for degraded yarns was found using the t-test ($P > 0.05$). In contrast, the RC decreased from 46% to 23% after 70 days of degradation. The RC of TAFe degraded for 28, 42, 56 and 70 days showed significant difference from the 0-day aged TAFe ($P < 0.001$). These results

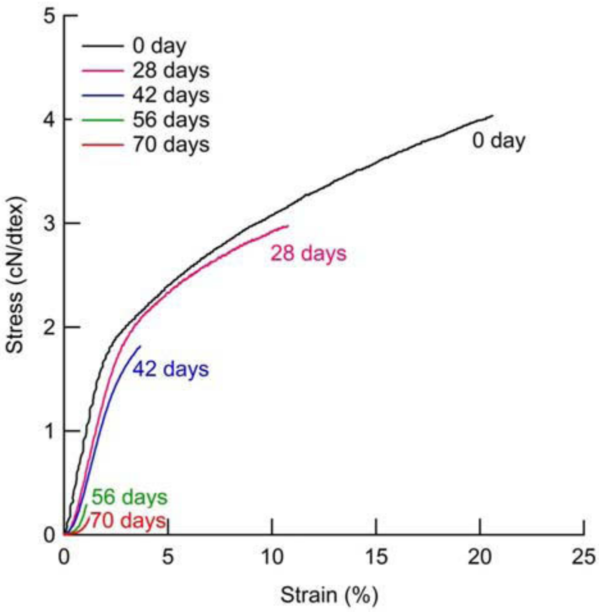

Figure 5: Tensile stress-strain curves of the TAFe yarns after the degradation (0, 28, 42, 56 and 70 days).

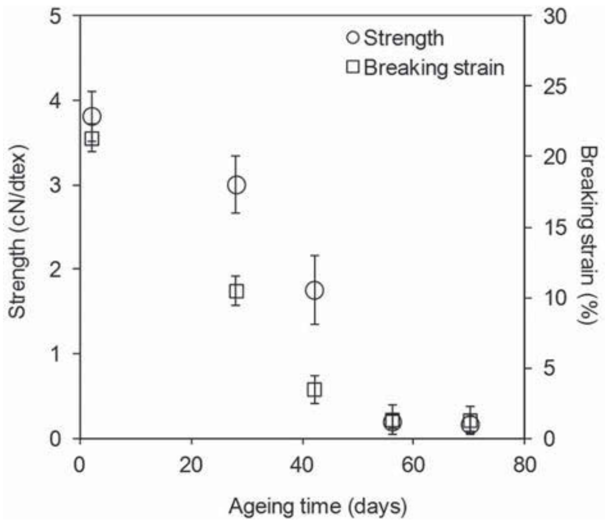

Figure 6: Strength and breaking strain for the TAFe yarns depending on the period of degradation. The data points represent averages and error bars show the standard deviations.

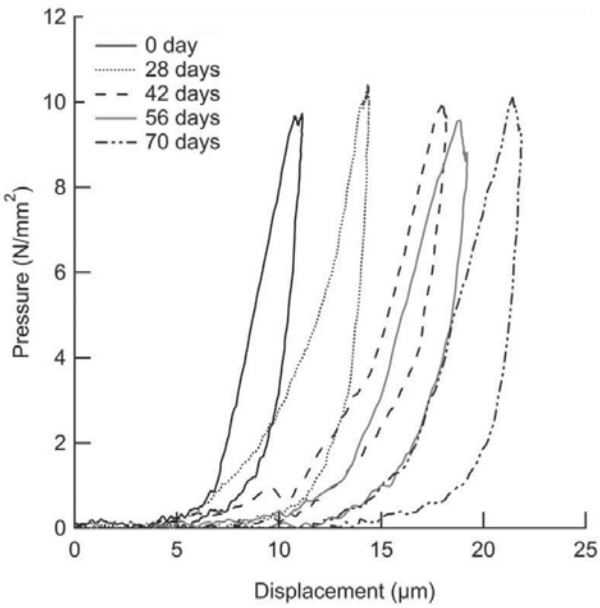

Figure 7: Compression stress-strain curves of the TAFe yarns after during degradation (0, 28, 42, 56 and 70 days).

indicate that the RC described the compression test better than δD in this experiment. By measuring the RC of yarns, it was found that the TAFe yarns tended to become brittle and fragile as degradation progressed.

In order to clarify the detailed relationship between the tensile and compression properties for the TAFe yarns, the value of compression resilience RC is plotted as a function of the tensile breaking strain and a trend line was added in Figure 9. As the compression test evaluates yarn resilience before ultimate breaking, tensile breaking strain is more adequate to describe the value of RC than ultimate strength. It was shown that the RC of TAFe yarns decreased as the tensile breaking strain of TAFe yarns decreased during the 70-day ageing period and a correlation between RC and breaking strain exists. On the basis of this experiment, it is expected that the tensile breaking strain could be estimated by conducting the compression test. In Figure 9, the mechanical properties of yarns were categorised into "Strong", "Weakened" and "Poor". The values delimiting categories were chosen on the basis of the actual experimental data for RC values.

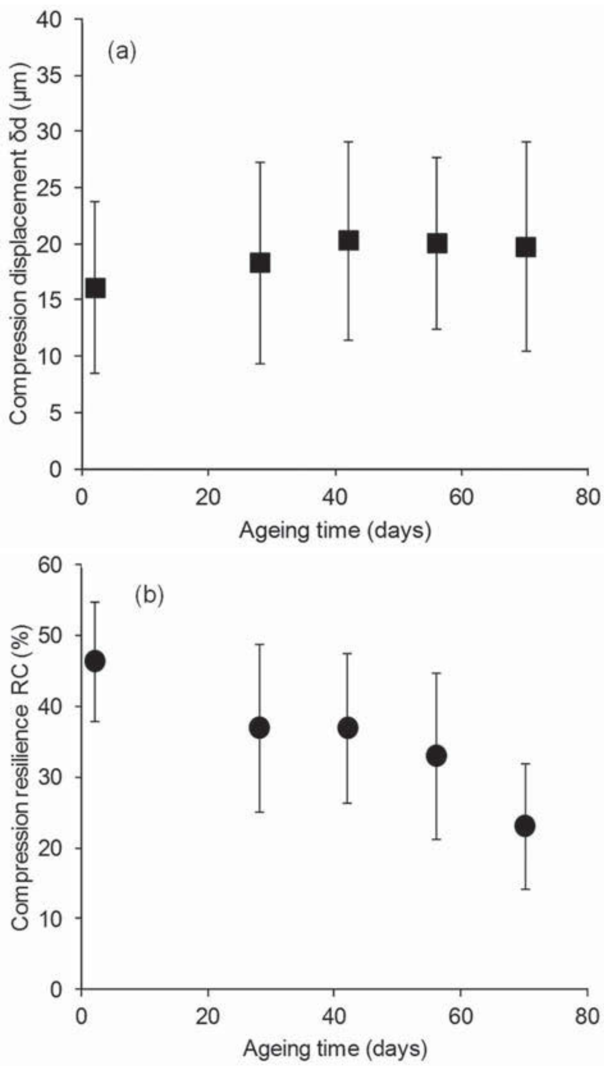

Figure 8: The result of displacement (a) and compression resilience (b) for TAFe yarns. The data points represent the averages and error bars show the standard deviations.

The non-degraded TAFe yarn samples are classified as "Strong". Their RC and tensile breaking strain values were more than 42% and 20%-25%, respectively. As for the "Weakened" state of TAFe yarns aged between 28 to 42 days, approximately between 34 to 42% of the RC was accompanied by 4-10%, on average, of the tensile breaking strain. In this case, while the yarns do not break immediately, it is necessary to take care of the environmental conditions by controling temperature and relative humidity and avoid further deterioration from careless handling. Finally, the yarns degraded for more than 56 days were in a "Poor" state. These yarns, with tensile breaking strain below 2%, had less than 34% RC. At this stage, since the yarns are easily pulverised by picking up with tweezers, it is very hard to measure their tensile strength. Accordingly, these results suggest that even if it is impossible to measure the tensile properties of yarns due to the

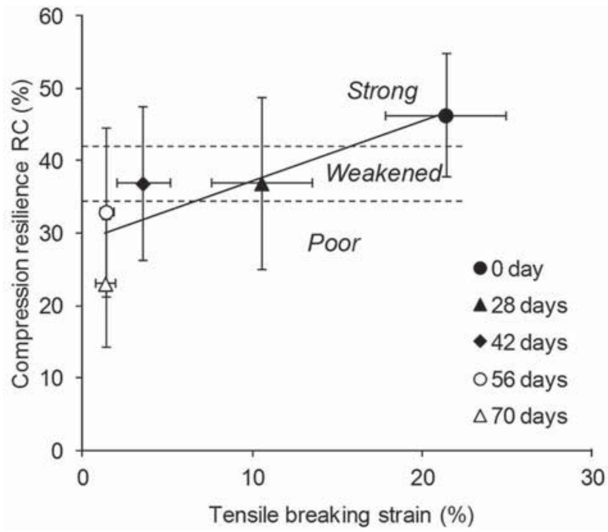

Figure 9: Relationship between tensile breaking strain and compression resilience for TAFe yarns. The physical properties of yarns were categorized into "Strong", "Weakened" and "Poor". The data points represent averages, and error bars show the standard deviations. A trend line was added.

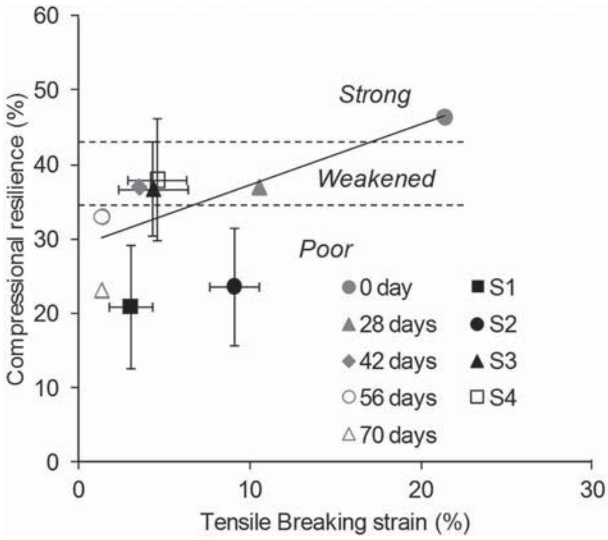

Figure 10: Relationship between tensile breaking strain and compression resilience for historic *Suga*-yarns (S1, S2, S3 and S4). The data points represent the averages and error bars show the standard deviations.

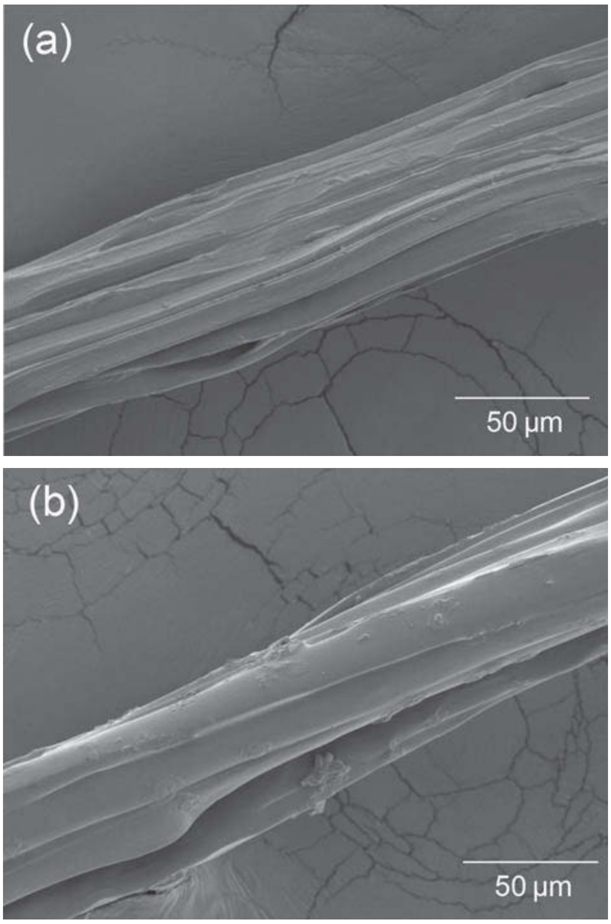

Figure 11: SEM micrographs of the TAFe yarn degraded for 70 days, before (a) and after (b) compression test.

“Poor” condition, the compression test may be used as an alternative.

In order to confirm the applicability to the historic *Suga*-yarns, the tensile and compression tests were performed for four original samples: S1, S2, S3 and S4. Figure 10 shows the relationship between the tensile breaking strain and the compression resilience RC. Every historic *Suga*-yarn exhibited approximately 20%-40% of the RC and less than 7% of the tensile breaking strain. By comparing the tensile and compression properties of TAFe (Figure 9), it was found that the historic *Suga* yarns S3 and S4 tended to have mechanical properties similar to those of TAFe yarns degraded between 28-42 days, categorized as “Weakened”. These yarns might degrade to the state “Poor”, so it is vital to focus on preventive conservation to avoid further degradation. S1 and S2 were categorized as “Poor” and yarns S1 flaked by just touching them with tweezers.

In contrast, S2 in the “Poor” category did not exactly correspond to the model samples with regards to breaking strain. Rather than being in “Poor” condition, it was close to “Weakened” because the mechanical state of S2 is relatively better than that of model yarns degraded for 56 days. In the case of real objects, such small variations would inevitably be encountered by conservators or conservation scientists.

Based on both experimental data and observation, it should be possible to estimate the mecanical condition of objects. The physical condition of historic *Suga*-yarns could be investigated by compression testing regardless of how much yarn becomes powdered. At present, it is still necessary to measure tensile properties in combination with compression properties in order to more acurately determine the mechanical condition of yarns. It is planned to continue the research to determine the characteristics of model yarns degraded under various conditions.

SEM was used to observe the changes in yarn surface morphology at the microscopic level, after compression testing. Figure 11 shows SEM micrographs of the surface of a 70-day aged TAFe yarn before and after compression. There is no apparent difference in the surface morphology of the yarn, which was typical of all the tested yarns. It is considered that the compression test, conducted using the KES-G5S is practically non-destructive.

4 Conclusion

To propose a practically non-destructive evaluation method using the KES compression tester with small samples, artificially degraded model raw silk yarns dyed with tannic acid and iron mordant were measured using both tensile and compression testers. It was found that as the tensile breaking strain declined with degradation, the RC decreased. It is clear that there is a relationship between tensile tests and compression tests. As a new approach, compression testing is thus useful when the amount of yarn is limited and/or it is difficult to measure tensile properties of degraded yarns. SEM observations showed there was no influence on yarn surface morphology after compression testing. It was thus confirmed that the compression test is suitable as a non-destructive evaluation method.

Using the same method and characteristic values as for the model yarns, the mechanical state of historic *Suga*-yarns was categorised. Although it is also vital to estimate the tensile properties in order to more accurately determine the mechanical condition of yarns, it was shown that the mechanical condition of historic *Suga*-yarns can be evaluated by performing compression tests.

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