DEVELOPMENT OF ENVIRONMENT CONTROLLABLE MICRO MECHANICAL TESTING MACHINE AND THE INFLUENCE OF VACUUM ON FRACTURE AND FATIGUE IN ARAMID SINGLE FIBER

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ABSTRACT

To perform micro mechanical tests including fatigue in microelements, a specially designed fatigue testing machine has been developed. The testing machine was designed to perform fatigue tests in microelements under simple- or three-point bending as well as under uniaxial loading. The testing machine is equipped with an environmental chamber to control the testing environment, and thereby the testing can be done in controlled environments such as vacuum or (humid) gases. Using the system, quasi-static and fatigue tests on aramid single fibers (Kevlar 49, diameter: about 13 μm) were performed. The tests showed that the strength of the aramid fibers is strongly influenced by environment. Water absorption and vacuum conditioning decrease the quasi-static fracture strength. Under fatigue loading, the aramid fiber has excellent properties compared with metallic materials, showing gentle slopes in *S-N* curves, although large scatter bands exist. The fatigue strength in vacuum is higher than that in air. The fiber breaks with fiber splitting. The fiber surface damage induced by fatigue loading in air and in vacuum was examined by using an atomic force microscope.

INTRODUCTION

Mechanical evaluation of micromaterials, or micro mechanical parts is one of the most important issues to be clarified. Typical small mechanical parts or microelements are the mechanical parts of future micromachines. The others are high-strength and high-elastic-modulus advanced fibers that are used for making rope or are reinforcement for

composite materials. As for microelements for micromachines, recent investigations have preferentially aimed at the processing methods. However, to develop a reliable micromachine in a service operation, mechanical properties including fatigue and wear response of the microelements must be understood. This will help to establish design criteria and life assessment methods for micromachines. As for advanced fibers, the mechanical properties of the fiber itself are extremely important [1-3], because the mechanical properties of a composite material are dependent on the properties of matrix, fiber and fiber/matrix interface. Of these, the longitudinal elastic modulus and fracture strength are strongly dependent on the properties of the fiber reinforcement [1]. Therefore, the fracture strength and fracture mechanism of the fiber itself must be determined, in order to better understand the performance of composite materials structures.

Besides the above considerations, micro mechanical components' properties are sensitive to the surrounding environment, because the surface to volume ratio increases with a decrease in the size of the component [4, 5]. This means that environmental strength evaluation is an extremely important research area. For example, quasi-static fracture and fatigue strengths of unidirectionally reinforced alumina fiber reinforced aluminum in air were decreased from that in vacuum. This was attributed to a decrease in fiber strength in air from that in vacuum due to water molecules in laboratory air [1, 3].

As for micro mechanical testing, the difficulties that we face are how small loads are accurately applied to micromaterials and how they are to be handled. Johansson and Schweitz [6] developed a testing machine that enables quasi-static simple bending tests as well as tensile tests [7] for a microelement. Tests can be conducted in a scanning electron microscope, and thereby *in situ* monitoring of testing sequence is possible. In their system, small loads are applied by a piezo-electric actuator. Piezo-electric actuator driven testing machines were also developed in ETH [8] and Toyota [9]. Other drive methods to apply small loads to microelements are DC motors [10] or electro-magnetic actuators [11].

However, these testing machines preferentially aimed at quasi-static tests and have scarcely been applied to fatigue testing: fatigue behavior is one of the most important issues in micro electro mechanical systems [4, 12-14]. Komai et al. [14] have developed a specially designed testing machine for microelements. Small loads can be applied to microelements using an electromagnetic actuator, and thereby mechanical testing including fatigue in microelements is possible. A drawback of the testing machine, however, is that testing in a controlled environment such as vacuum or humid gases, is impossible.

In this investigation, the authors have developed a micro mechanical fatigue testing machine equipped with an environment chamber. Using this new testing machine, micro mechanical fatigue tests were conducted in advanced aramid fibers. Special attention was paid to environmental effects of vacuum on the fracture and fatigue behavior. Nanoscopic fiber surface damage was evaluated by using an atomic force microscope [15], and the fracture mechanism and the influence of vacuum on the fracture behavior are discussed.

ENVIRONMENT CONTROLLABLE MICRO MECHANICAL FATIGUE TESTING MACHINE

The authors' group [14] has already developed a testing machine, which enables mechanical testing including fatigue of a microelement. Small loads (load range: 0.1 mN - 5N, accuracy: 0.02 mN) are applied by means of an electromagnetic actuator, and the associated displacement is measured with a differential transformer. The operation of the testing machine is computer controlled, and real-time data acquisition and data analyses are thereby possible. Although precision micro mechanical testing can be conducted using the developed system, drawbacks are that the loading frequency is limited to only 0.1 Hz and that it was designed to operate only in laboratory air. However, as is discussed before, the effects of environments on mechanical properties become extremely important in micromaterials, and therefore, the mechanical evaluation should be performed in a controlled environment.

Considering these, the authors have developed a micro mechanical fatigue testing machine that is equipped with an environmental chamber. Micro mechanical tests including fatigue are thereby possible in a controlled environment such as vacuum (10^{-4} Pa) and various (humid) gases. The environment chamber is evacuated by a diffusion vacuum pump (200 L/s) and a rotary pump (160 L/min). Small loads (maximum load: ±5 N, maximum displacement: ±1.25 mm) are applied by an electro-magnetic actuator, and fatigue tests can be conducted at higher stress cycle frequencies up to 20 Hz. The testing machine will accomodate three-point bending simple bending, and axial load type testing. The applied load is measured by a precision type load cell with a full scale of either 5 N or 1 N. The displacement of the loading point is measured with a linear differential transformer. The testing machine can operate under either a load control or displacement control. The testing machine is also equipped with a programmable function generator, and a desired loading wave form can be applied to a sample, including sinusoidal wave, saw-tooth wave, and positive and/or negative pulse wave forms.

In the case of bending tests, the sample must be loaded at the center of a microbeam under three-point bending, and at the middle part in the width direction under simple bending. For this purpose, the testing machine is equipped with a top viewer, or an optical microscope, for monitoring specimen alignment. In the case of bending tests, force can be applied to a microelement by a stylus positioned at the end of the actuator, which is a spherical diamond tip of 20 μm radius and 60° cone angle. For three-point bending, a specially designed jig to support a microelement is used: two roller support (diameter: 0.6 mm) are used and the spacing between the roller can be varied as a test requirement.

FRACTURE AND FATIGUE BEHAVIOR OF ARAMID SINGLE FIBER

Experimental Procedures

The aramid fiber used was Kevlar 49 manufactured by du Pont, USA. Single fiber quasistatic tensile tests were conducted, following the recommended testing procedures as described in ASTM D3379/JIS R7601. Figure 1 illustrates the single fiber tensile test

specimen. The fiber was glued to a tab made of polyester thin film, in order to easily handle the small diameter fiber. Using the developed testing machine, tensile tests in laboratory air and in vacuum (4 - 5 ∞ 10^{-3} Pa) were conducted on the fibers preconditioned in laboratory air at a displacement rate of 3.5 ∞ 10^{-2} mm/s. The fibers were stored in laboratory air for about three to four years before conducting the mechanical tets. The load cell used here had a capacity of 1 N.

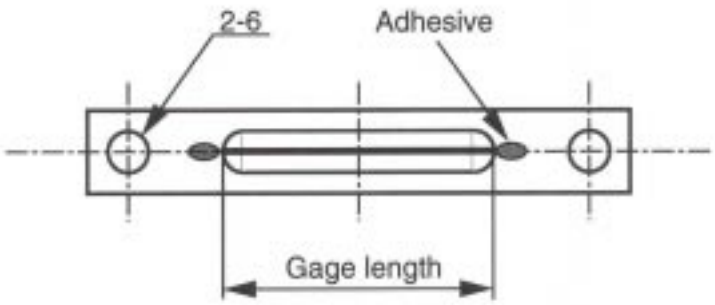

Figure 1: Single fiber tensile test specimen

Fatigue tests were also conducted in air and in vacuum (1 - 2 ∞ 10^{-3} Pa) under a load control at a stress ratio of 0.2 and a frequency of 5 Hz. Fiber damage was observed with a field-emission, high-resolution scanning electron microscope (S4500, Hitachi Ltd., Japan). For some fibers, nanoscopic fiber surface damage was closely examined with a scanning atomic force microscope (AFM; NanoScope III and multi-mode SPM unit, Digital Instruments Co., USA).

Out Gassing Behavior of Fiber

Figure 2 illustrates the mass change of the Kevlar 49 fiber that was conditioned in vacuum for 4 h. The change was measured in laboratory air just after the fiber was taken from the vacuum chamber to laboratory air. The ordinate adopted is the fiber mass normalized by the mass measured before vacuum conditioning. An increase in the mass is attributed to re-absorption of moisture, and it is reasonable to asuume that this moisture uptake reached the same equilibrium level as the levlel in the fiber before vacuum exposure. Therefore, the outgassing of the fiber can be obtained from re-absorption data, and it was about 3 to 4 % to the total mass of the fiber. The amount of outgassing measured here was almost equal to the equivalent water sorption in humidified air. Others reported moisture uptakes of 3% (RH50%, 30°C) [16] to 4.5% (RH55%, 23°C) [17].

Effects of Vacuum on Single Fiber Tensile Strength

Aramid fibers absorb water, and such absorbed water degrades the axial [2, 18] as well as lateral mechanical properties [19]. The longitudinal tensile strength and elastic modulus are smaller in the fibers preconditioned in hot water at 80°C than those conditioned in laboratory air [2]. The failure is associated with longitudinal splitting, and the amount of fiber splitting is more conspicuous in the fibers conditioned in hot water than in the fibers conditioned in air.

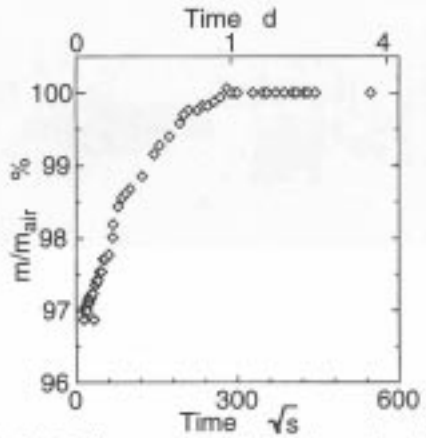

Figure 2: Changes in mass of Kevlar 49 fiber as a function of time after the environment was changed from vacuum to air.

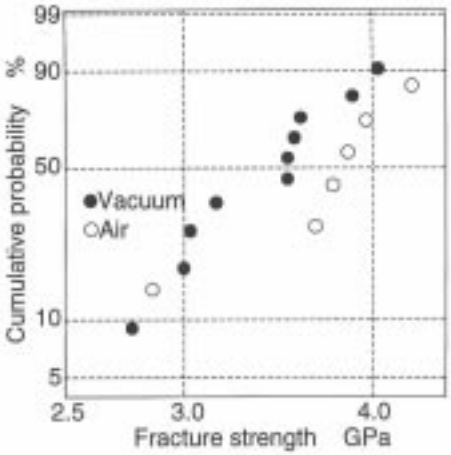

Figure 3: Weibull plots of the tensile strength of Kevlar 49 fiber tested in laboratory air and in vacuum.

Figure 3 illustrates an example of Weibull plots of the tensile strength of fibers conditined in laboratory air and in vacuum. In contrast with the results for fibers conditioned in hot water, the tensile strength in vacuum was lower than that in air. The fiber failure was associated with fiber splitting, and an amount of fiber splitting in vacuum was larger than that in air. These results suggest that by conditioning in vacuum, water and/or other gasses diffused out of the fibers, and then they became susceptible to fiber splitting, resulting in a decrease in fiber strength.

Effects of Vacuum on Single Fiber Fatigue Strength

Figure 4 shows the *S-N* curves of Kevlar 49 single fiber conducted in air and in vacuum. The left-pointing arrows on the low-cycle end of Figure 4 indicate that the sample failed during fine adjustments of loading levels. First, the *S-N* curves have a large scatter band, similarly to the quasistatic tensile strength. Second, the slope of the *S-N* curves is gentle compared with those of metallic materials, indicating that the Kevlar 49 fiber has excellent fatigue properties. Finally, the fatigue strength of fiber in vacuum was higher than that of fibers in laboratory air. However, as is shown in the previous section, efects of environment on tensile strength was the oposite. Namely, the tensile strength of fibers tested in air was higher than that of fibers tested in vacuum.

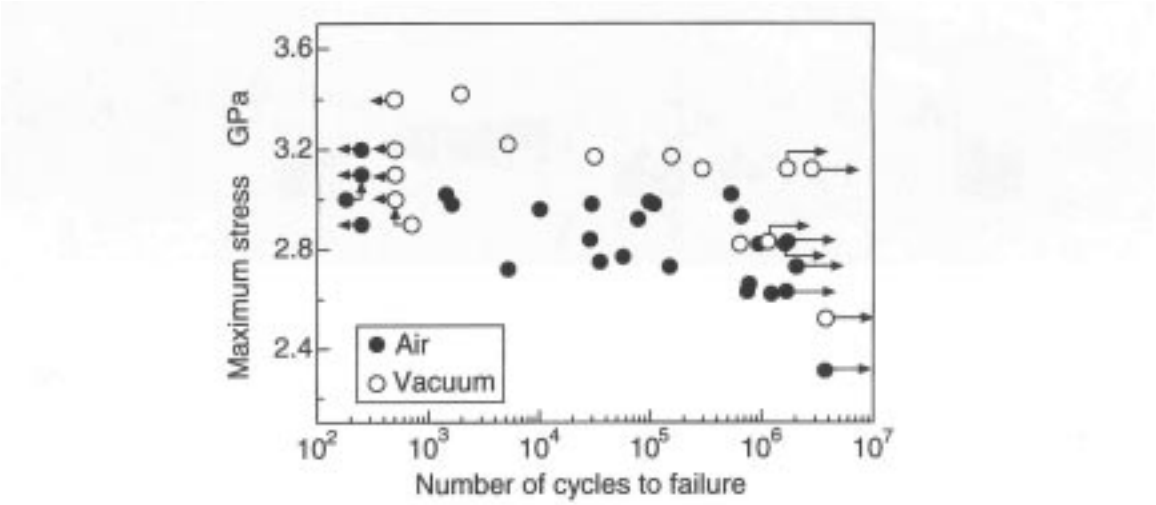

Figure 4: *S-N* curves of Kevlar 49 fiber in laboratory air and in vacuum.

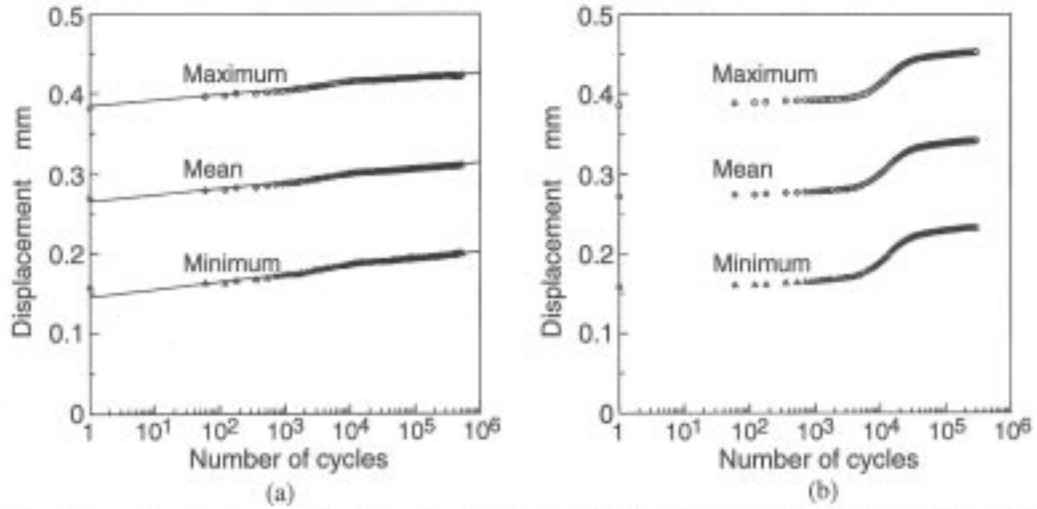

Figure 5: Changes in displacement as a function of stress cycles during single fiber fatigue tests in (a) laboratory air and in (b) vacuum.

Figure 5 shows changes in displacement as a function of stress cycles. The displacement increased gradually in air, and its change was linear when plotted semi-logarithmically (Figure 5(a)). However, in vacuum (Figure 5(b)), the displacement abruptly increased at a certain stress cycle. This abrupt increase in displacement was observed in all tests conducted in vacuum, not in air.

Similarly to the case of tensile tests, the fiber broke with fiber splitting. In air, the splitting length at breaking point was about 60 μm, some of which got tangled and the tip of which branched into several parts (see Figure 6(a)). In contrast with these, the shape of the splitting in vacuum was simple, and no tangled one was observed (Figure 6(b)). In addition, the length of the splitting at breaking point was more than 200 μm: the splitting length relating to fiber breakage in vacuum is longer than that fatigued in air.

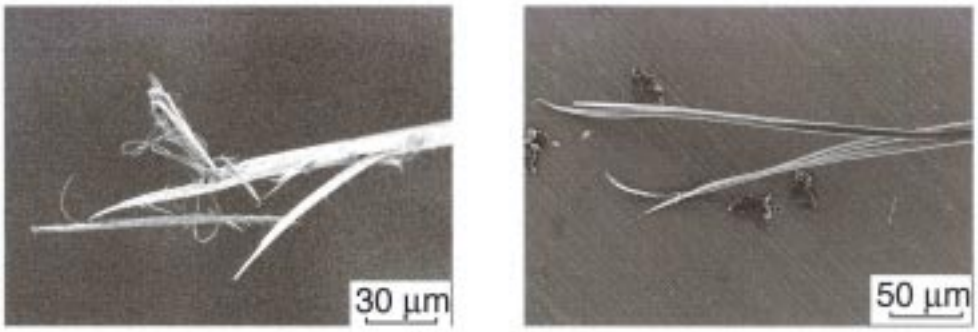

Figure 6: Fracture morphology of Kevlar 49 fiber fatigued in (a) air (maximum stress: 2.73 GPa, $N_f = 1.49 \times 10^5$) and in (b) vacuum (maximum stress: 3.42 GPa, $N_f = 1.98 \times 10^5$).

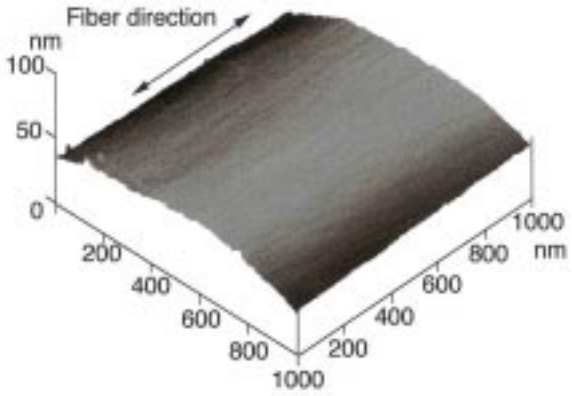

Figure 7: AFM image of virgin fiber surface of Kevlar 49, which was preconditioned in air.

Nanoscopic Fatigue Damage Evaluation by AFM

Micromaterials such as the Kevlar fibers used in this investigation are smaller than ordinary mechanical parts and have characteristic dimensions on the order of μm. Hence, the damage that causes fracture and degradation in strength is localized to extremely small areas, compared with traditional mm sized mechanical parts. Therefore, higher magnification, or nanoscopic surface damage evaluation [15] is required, instead of traditional μm-order damage evaluation by using, for example, a scanning electron microscope.

Figure 7 [2] illustrates the contact mode AFM image of a Kevlar 49 fiber conditioned in laboratory air at room temperature. The image was taken before mechanical testing. Typical longitudinal fibril structure [20] can be seen, indicating fiber molecules align along the fiber axis, or longitudinal direction. However, surface roughness is very small. The R_a value calculated was 0.67 nm.

Figures 8 and 9 respectively illustrate the AFM images of fiber surface, fatigued in air and in vacuum. On the surface of the fiber fatigued in air, “nodule” type fiber surface damage can be seen. The size of nodules ranged from about 30 to 50 nm, and some large nodules were about 100 to 200 nm. The ratio of large nodules tended to increase with stress cycles (Figure 8). These nodules are considered nuclei that will lead to splitting.

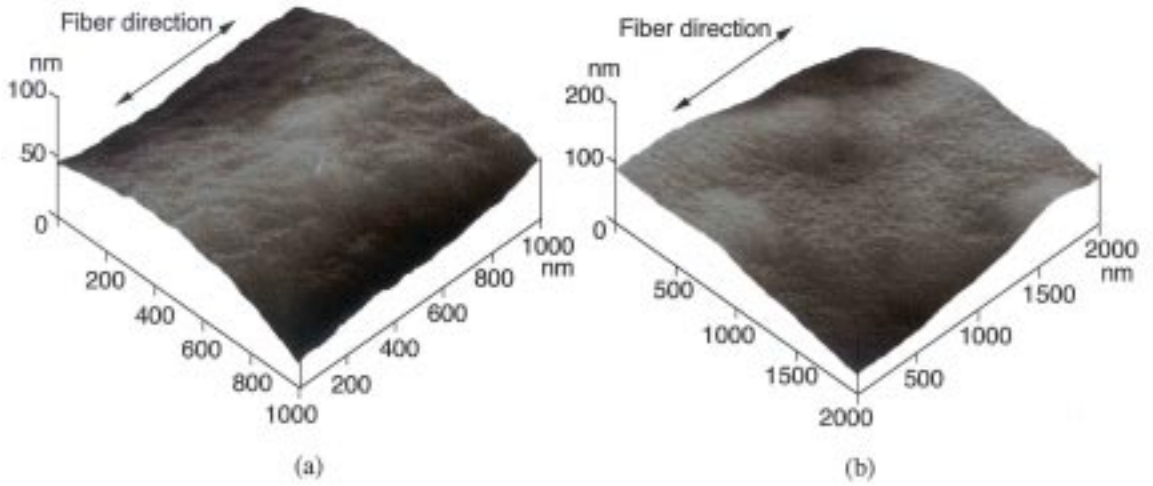

Figure 8: AFM image of Kevlar 49 fiber, fatigued in laboratory air. The images were taken the fatigue test being interrupted at (a) $n = 1 \times 10^4$ cycles and (b) $n = 5 \times 10^4$ cycles.

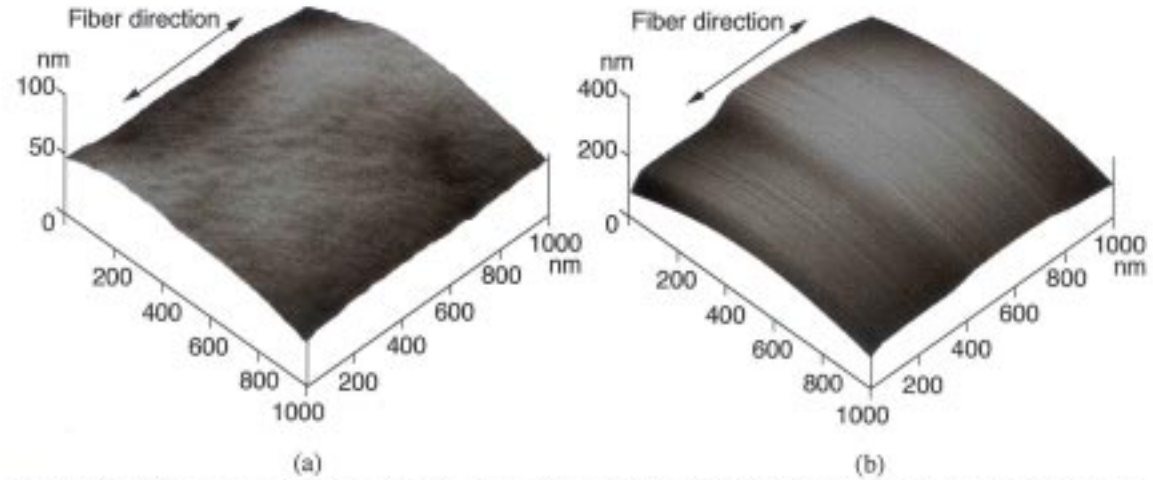

Figure 9: AFM image of Kevlar 49 fiber, fatigued in vacuum. The images were taken the fatigue test being interrupted at (a) $n = 1 \times 10^3$ cycles and (b) 1×10^4.

Similarly to the fatigue damage observed in air, the fiber surface fatigued in vacuum consisted of “nodule”, when the number of fatigue cycles was less than the stress cycles above which an abrupt increase in displacement was observed (Figure 5(b)). However, when the number of cycles was greater than the cycles where the abrupt increase in displacement occurred, the fiber surface looked different (Figure 9(b)). The cross section normal to the fiber axis became extremely smooth, and no trace in fibril structures could be seen. Along the fiber direction, large “undulations” could be seen. The abrupt increase in displacement and associated nanoscopic fiber surface changes in morphology may relate to rearrangement of molecular structures. In vacuum, fatigue loading promotes the rearrangements in molecular structures in fiber, resulting in a large increase in displacement and improvement in fatigue strength. However, further investigation is required in the mechanisms of fatigue and how the vacuum environment affect the fatigue strength.

CONCLUSIONS

A specially designed environment-controllable micro mechanical fatigue testing machine is presented that is capable of performing quasi-static and fatigue tests in μm sized micromaterials such as high-strength and high-elastic modulus fibers. Using this testing machine, micro mechanical fatigue tests were conducted on Kevlar 49 single fibers. The fracture mechanisms, in particular from nanoscopic points of views based upon atomic force microscopy were discussed. The investigation yielded the following conclusions.

1. By using the developed micro mechanical fatigue testing machine, the fatigue tests in small diameter single fiber (diameter: 12 μm) can be conducted in a precise manner.
2. Kevlar 49 fiber shows lower tensile strength when conducted in vacuum than in laboratory air.
3. Kevlar 49 fiber has excellent fatigue properties, although there is large data scatter. The *S-N* curve has a gentle slope compared with metallic materials.
4. The effect of environment on fatigue strength is contrary to the effect on tensile strength. The fatigue strength in vacuum is higher than that in air. Fibers break with fiber splitting, irrespective of environment. However, the splitting length at failure is smaller in air than in vacuum, and the shape of the splitting in air is more complicated.
5. Nanoscopic fiber surface damage is successfully imaged by an atomic force microscope. Fatigue loading in air causes nanoscopic “nodule” type fiber surface damage, which are considered nuclei for splitting. The fiber surface fatigued in vacuum consisted of “nodule”, when the number of fatigue cycles was less than the stress cycles above which an abrupt increase in displacement was observed. However, when the number of cycles was greater than the cycles where the abrupt increase in displacement occurred, large “undulation” is observed along the fiber axis, and the cross section normal to the fiber axis becomes extremely smooth, and no trace in fibril structures can be seen.

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