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# A NOVEL METHOD FOR THE MEASUREMENT OF THERMAL EXPANSION OF THIN FILM USING LASER SCANNING CONFOCAL MICROSCOPY

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## ABSTRACT

For the fabrication of Micro Electro-Mechanical System (MEMS), film fabrication process combined with photolithography is thought to be one of the fundamental techniques. However, the physical properties of films and membranes strongly depend on their processing routes. For designing MEMS as engineering products, the control of physical properties such as the coefficient of thermal expansion (CTE) and hardness are important. However the CTE measurement of thin films is very difficult because the magnitude of displacement to be measured is about 1/10000 of the thickness of the specimen, i.e., of the order of nano-meter for specimens with micron-meter thickness. In this study the measurement of CTE of several ten micron meter-thick metal thin film based on an optical interference was attempted using a Laser Scanning Confocal Microscope (LSCM). The specimen was set between two alumina single crystal plates as optical flats. He-Ne laser is reflected from the bottom surface of the upper optical flat (which is transparent) and the top surface of the lower optical flat, and these two rays interfere either constructively or destructively, resulting in the formation of interference fringes. It was shown that by counting the number of fringes observed using LSCM, the expansion of a specimen as thin as a few-tenth micron meter can be detected.

## KEY WORDS

Coefficient of thermal expansion, Fizeau interferometer, MEMS

## INTRODUCTION

In view of the industrial use of materials at a temperature range that is different from the processing temperature, many problems such as distortion and cracking arise due to the thermal expansion. Especially in a component composed of several materials having the coefficient of thermal expansion (CTE) difference the mismatch of length occurs, resulting in serious residual stress and the change of its shape. In order to minimize such problems through the process-route control and the component design, the CTE data of the materials is most fundamental information. For such purpose, various techniques has been employed for the measurement of thermal expansion, as reviewed by James et al. [1], and data has been accumulated.

Recently, a new class of machinery industry, micro-electro-mechanical systems (MEMS) industry, have been growing [2]. As these machines have their typical dimensions of the order of microns, their processing through micromachining technology is based on film fabrication techniques such as chemical vapor deposition (CVD) and physical vapor deposition (PVD) combined with photolithography techniques, which are far different from that for the conventional machines such as the cutting, drilling and shaving of the bulk materials from the melt. However, it is not clear whether the materials produced through such processing routes have the same physical and mechanical properties as that of bulk materials. In the materials processed by CVD or PVD, a large amount of defects are introduced during processing, which may result in unexpected change of properties such as the thermal expansion [3]. Moreover, the thermal expansion is one of the important driving forces for the active MEMS devices like a micromirror array, which will be applied for optical beam steering, optical data interconnection, real-time image recognition, and aberration correction [4]. The micromirror array is fabricated through a commercial Complementary Metal Oxide Semiconductor (CMOS) process because it is integrated with piezoresistive deflection sensors and CMOS switching circuit for controlling the heating by applying electrical power to individual mirror deflection. The difference in thermal expansion between the Al and $SiO_2$ layers consisting of the supporting flexures cause the curl of the flexures, resulting in the motion of mirror. Because of the complex flexure geometry, various approaches have been applied such as a bimorph beam theory and finite element techniques for modeling the static deflection of the mirror. In such modeling the material properties were assumed to be constant throughout the beam and with respect to time [4]. However, the CTE values may be different from those for bulk materials, and a large amount of defects introduced during processing may result in the unstable behavior of thermal expansion during the thermal cycle.

For extending the application area of MEMS devices, characterization of physical and mechanical properties of thin film materials is needed. Especially as the size of components is not a factor for the residual stress caused by thermal expansion, the technique for the thin film thermal expansion measurement is needed to establish not only to accumulate basic CTE data for designing but also to select a proper combination of material and processing route for producing durable MEMS components. However, the demand for the establishment of a standard technique for MEMS industry has not been fulfilled.

According to James et al., various techniques have been proposed for the thermal expansion measurement of materials [1]. Most of them are for the specimens with the size of the order of several mm or larger, and quite a few techniques have been proposed for micron-size materials. Tada et al. have proposed a method to estimate the thermal expansion of Si which is one of the constituent materials of multilayered cantilever beams composed of Si and $SiO_2$ layers by measuring the thermally-induced

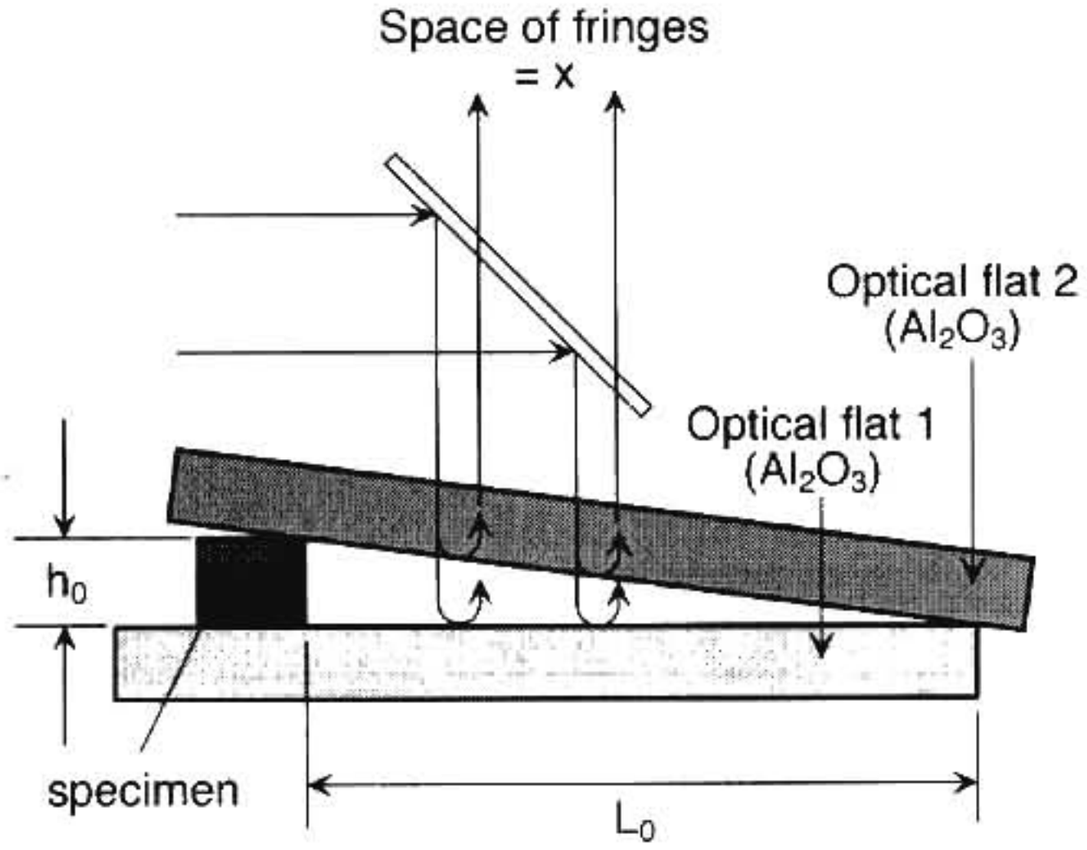



Fig.1 The experimental set-up.

curvature of beams at high temperatures [2]. This method enables to measure the thermal expansion of materials as thin as sub-microns, but the accuracy of the beam shape and the thickness of each layer are needed to be as high as possible, and the CTE of substrate material (in this case, $SiO_2$) is inevitable for the calculation. Other techniques based on the micromachining technique to realize a complex structure for the thermal expansion measurement have been proposed [5]. Although these techniques are suitable to understand the behavior of the materials and structures to be used in a specific case, it is hard to utilize for the accumulation of the thermal expansion data of materials processed by various techniques. The accuracy of the measurement by these techniques is commonly affected by the accuracy of the specimen dimension and shape, which are barriers to measure the CTE of small size specimens. X-ray diffraction (XRD) pattern is a powerful method for the estimation of the CTE by measuring the lattice parameters of crystalline materials because the method is free from the limitation by the specimen size [6]. However, the change of lattice parameter is not principally the same with the change of specimen size because the decrease or increase of the defects such as vacancies varies the specimen size. Moreover, XRD method is not applicable for materials having no crystal structure such as amorphous or bulk metallic glass. The CTE measurement in a microscopic scale by using the Atomic Force Microscope has been proposed [7]. This method is expected to measure the height of the surface step with good accuracy, but the thermal expansion of the supporting system affects the accuracy.

Methods based on various interferometric techniques such as Michelson interferometer, Fabry-Perot interferometer and others have also been proposed for the accurate measurement of thermal expansion. These techniques have advantage that little mechanically movable component is required, but needs to fulfill the common conditions such as the preparation of a high reflective surface and the measurement of height of specimens. For the measurement of the thermal expansion of small specimens, easy handling is inevitable; it is very difficult to polish the surface and to measure the actual size of the small size specimen. Moreover, it is impossible to make the specimens in parallel-piped shape with certain accuracy in size without a special apparatus such as Focused Ion Beam (FIB) system.

The present authors have proposed to apply an old-fashioned interferometric method, Fizeau interferometer, combined with a spatial carrier fringe pattern analysis for the easy measurement of the CTE by measuring the change of the height of small size specimens without measuring the size and other spatial configurations. In the present paper, a brief sketch of this method is presented, and the results of the measurement of the CTE of specimens with a dimension of the order of several ten μm. Some ideas for further applications will also be described.

## THE CONCEPT OF THE METHOD

Fig.1 describes the experimental set-up. Specimen is placed on a well-polished substrate (optical flat 1), and a transparent plate (optical flat 2) having well-polished surfaces is set as illustrated in the figure. Laser beam reflected by the optical flats form a spatial fringe pattern. It is obvious that the fringe pattern becomes narrow with increase in the angle between optical flats. The space of fringes, $x$, at room temperature, $T_0$, is expressed as follows;

$$x = \frac{\lambda}{2}\frac{L_0}{h_0} \qquad (1),$$

where $\lambda$ is the wave length of the laser beam, $h_0$ is the height of the specimen, and $L_0$ is the distance between the contact position of the optical flats and the contact position of the specimen and the optical flat 1. For the measurement only the laser beam from the top surface of the optical flat 1 and the bottom surface of the optical flat 2. Therefore, the thickness change of optical flats during the heating and cooling is not needed to be taken into account. The contact points among the specimen and optical flats are expected to be fixed during the heating

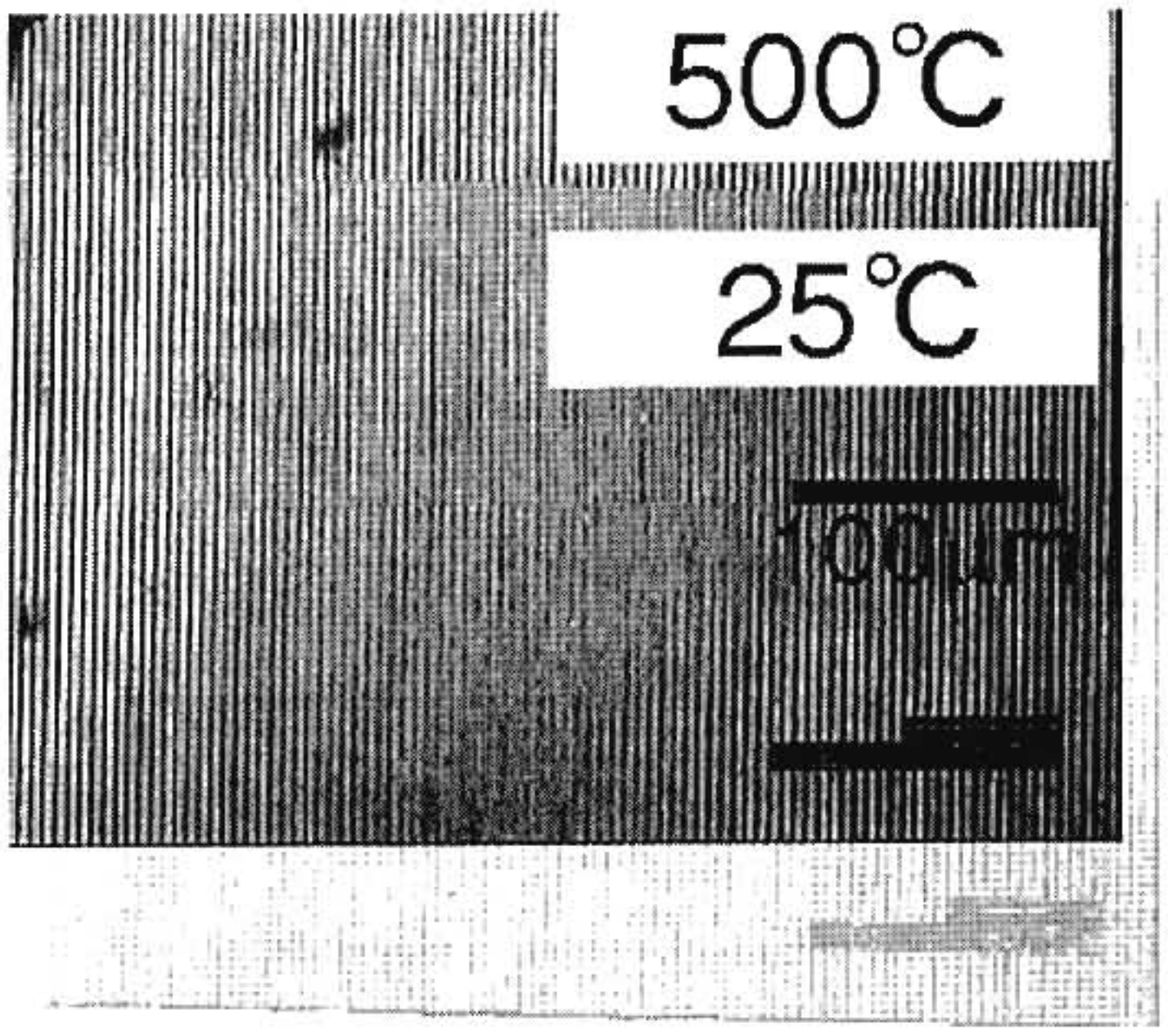



Fig.2 A typical pattern of interference fringes.

and cooling because the weight of the optical flat 2 results in friction at the contact points. According to the definition of CTE, $\alpha = \frac{l - l_0}{l_0}\frac{1}{T - T_0}$, where $l$ and $l_0$ are the specimen sizes at the temperatures $T$ and $T_0$, respectively. Then, by taking the thermal expansions of the specimen and optical flats into account, the space of fringes at a temperature, $T$, is expressed as follows;

$$x(T) = \frac{\lambda}{2}\frac{L_0\{1 + \alpha_B(T - T_0)\}}{h_0\{1 + \alpha_A(T - T_0)\}} \qquad (2),$$

where $\alpha_A$ and $\alpha_B$ are the CTE of specimen and optical flats, respectively. By combining eqs.(1) and (2), one can obtain the following expression;

$$\alpha_A = \frac{1}{(T - T_0)}\left[\frac{x(T_0)}{x(T)}\{1 + \alpha_B(T - T_0)\} - 1\right] \qquad (3).$$

By adopting $\alpha = \frac{l - l_0}{l_0}\frac{1}{T - T_0} = \Delta l / \Delta T \,/\, l_0$ for each temperature,

$$\alpha_A = \frac{1}{\Delta T}\left\{\frac{1 + \alpha_B \Delta T}{1 + \frac{\Delta T}{x(T)}\frac{\partial x}{\partial T}} - 1\right\} \qquad (4).$$

It is quite interesting that only the relative change of

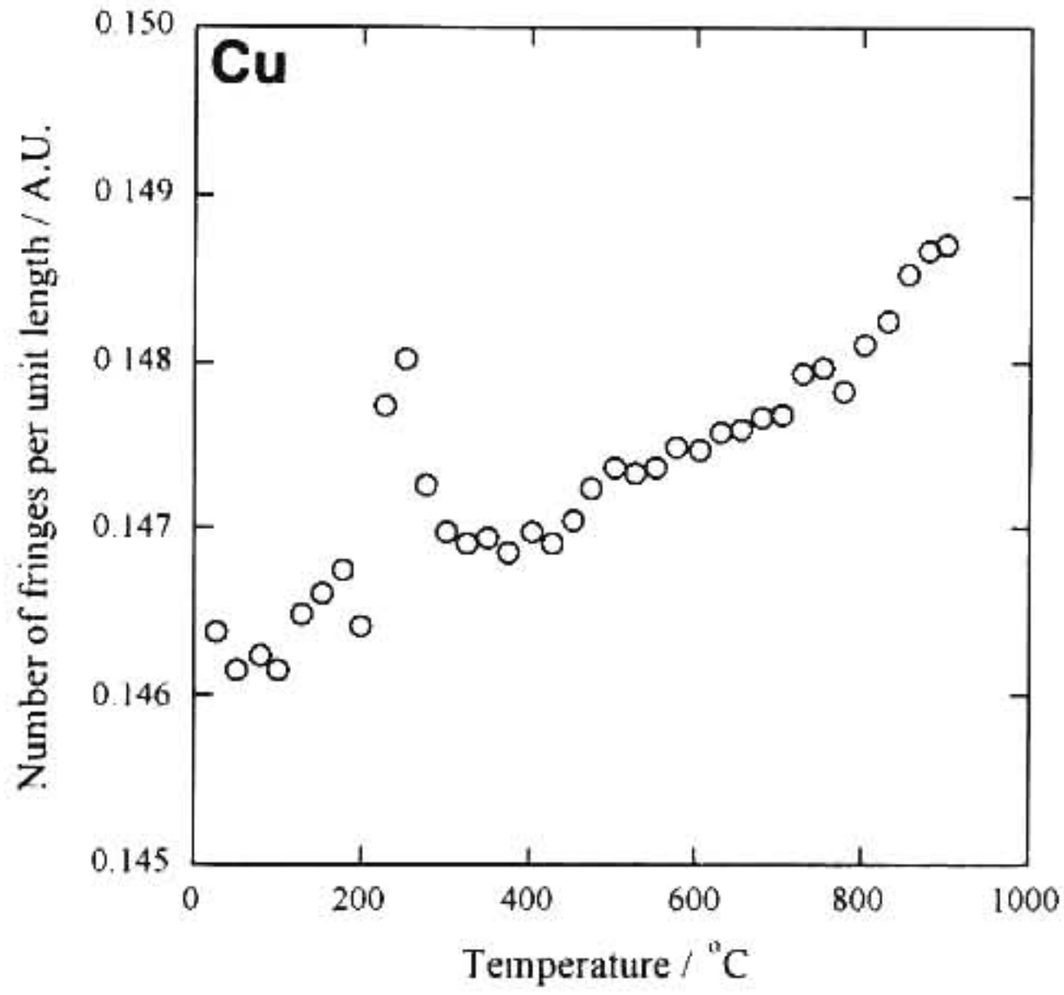



Fig.3 Temperature dependence of the number of fringes per unit length of the Cu specimen during heating.

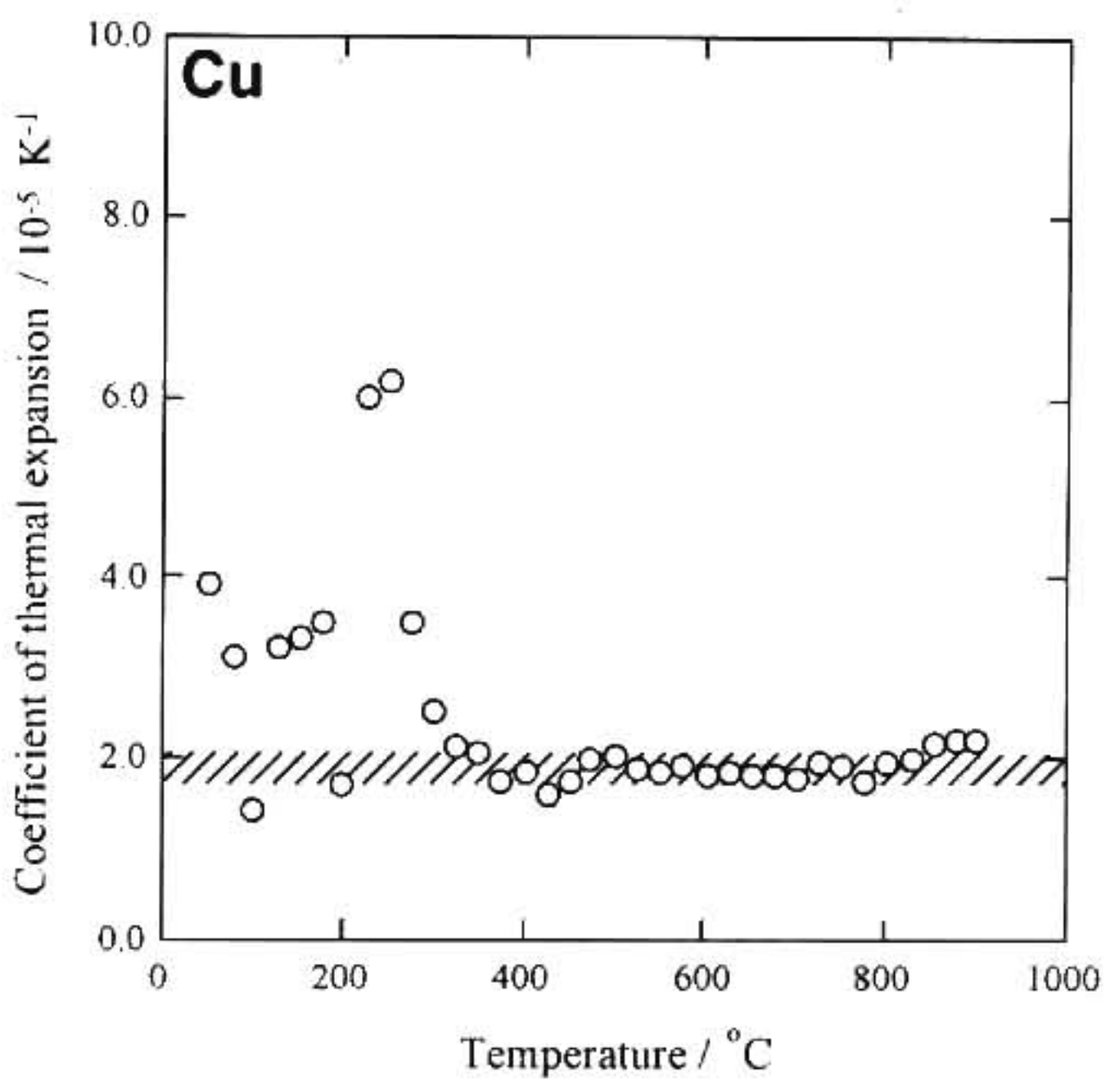



Fig.5 Temperature dependence of CTE of Cu specimen during heating, measured by proposed system.

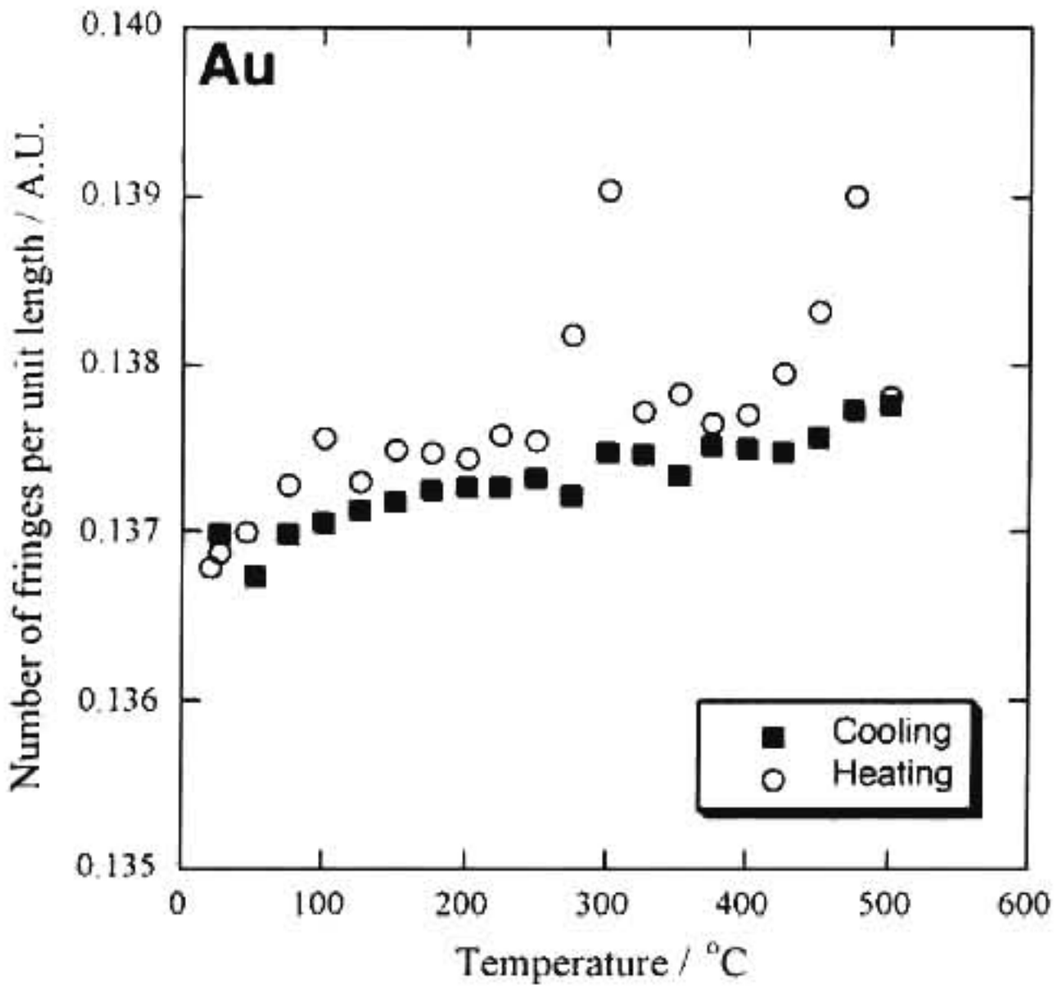



Fig.4 Temperature dependence of the number of fringes per unit length of the Au specimen during heating and cooling.

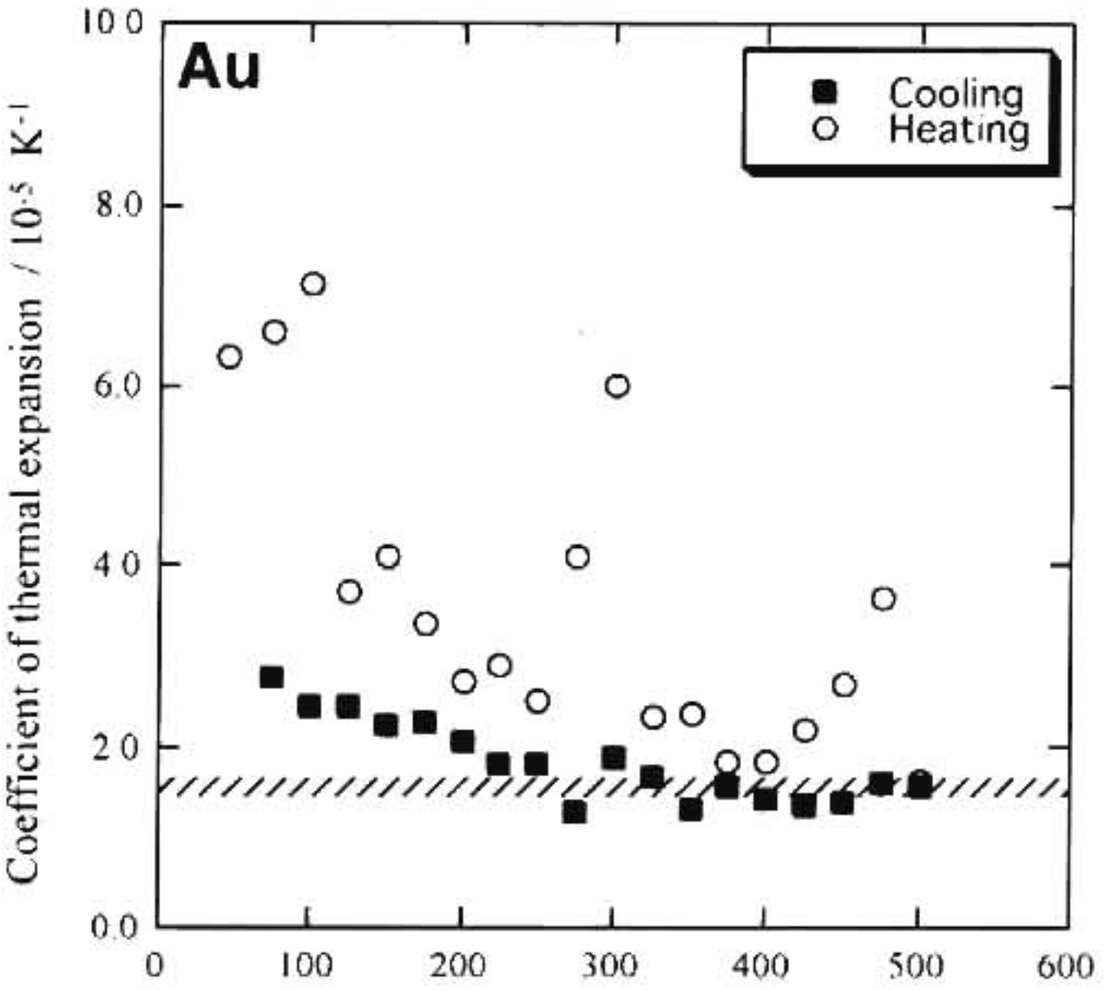



Fig.6 Temperature dependence of CTE of Au specimen during heating and cooling, measured by proposed system.

the space of fringes is needed for the estimation of the CTE in the proposed method; no information on either the specimen size or the location of specimen are required, and no uncertainty caused by the physical properties of surroundings are introduced. Only the CTE of the optical flats, $\alpha_B$, is taken into account. These are quite large benefits especially for the CTE measurement of small size specimens because with decreasing the specimen size it becomes harder and harder to prepare the specimen size accurately enough for the precise measurement of CTE.

## EXPERIMENTAL PROCEDURE

The measurements of the CTE of some metal specimens (Cu and Au thin specimens with thickness of about 50 and 200 μm, respectively) were carried out. $Al_2O_3$ single crystal (Sapphire) plates with C-axis normal to the flat surface were used as optical flats with a thickness of 0.5 mm and $\alpha_B = 5\times10^{-6} K^{-1}$, which has very small temperature dependence at the temperature range for the measurement in the present study. The measurement apparatus was set in a gold image furnace attached to a laser scanning confocal microscope (LSCM, type 1LM21H, Lasertech Ltd., He-Ne laser, λ=662.8nm). Various kinds of fringe patterns are expected to be observed because there are four flat surfaces for laser beam reflection. On this point LSCM has an advantage to detect only the fringe patterns formed by interference of laser beams reflected from the upper surface of the optical flat 1 and the bottom surface of the optical flat 2 near the contact position of these two optical flats. It is caused by the narrow depth of focus of LSCM, and only the fringe pattern of concern is observed while other fringe patterns are out of focus [8, 9]. Also the observation free from the speckle patterns is advantage.

Fringe patterns are recorded by a video recorder during heating and cooling at a rate of 300 K/min for Cu specimen and 50 K/min for Au specimen under a high purity Ar (99.9999%) flow atmosphere. The average space of fringes at each temperature was evaluated either by measuring the length for several ten fringes by scale, or by counting the number of fringes in a certain length by Fourier pattern analysis.

## RESULTS AND DISCUSSION

Fig.2 shows a typical fringe pattern. The periodicity of the fringe patterns is quite good, and also the difference in the space of fringes at different temperatures can be recognized. The number of fringes at a certain length at 500 °C is larger than that at 200 °C. In Fig.3, temperature dependence of the number of fringes per unit length of the Cu specimen during heating, which is reciprocal of the space of fringes, $x$, is shown. The number of fringes per unit length increases with increasing temperature during heating. There is a "jump" at around 200 - 300 °C. The similar jump was also observed in Fig.4 showing the temperature dependence of the number of fringes per unit length of the Au specimen during heating and cooling. The origin of these jumps is not clear, but this may not be due to the intrinsic properties of specimens but may be related to an unexpected deformation of the measurement apparatus such as a change in the position of Pt bed on which all of the measurement apparatus are set. The Pt bed is supported by four ceramic tubes each of which may change their length during heating and cooling. The difference between the thermal expansions of ceramic tubes may twist the position of the Pt bed, which may cause the sudden change in the measured value of the number of fringes per unit length.

As the initial size of the specimen and the location of the specimen are not identical, the value for the number of fringes per unit length is different in each experiment as shown in Figs.3 and 4. However, the absolute value for the number of fringes per unit length is not required for the estimation of the CTE in the proposed method as shown in eqs.(3) and (4). The temperature dependence of CTE for Cu and Au specimens are shown in Figs.5 and 6, respectively, estimated using eq.(3). In Fig.5 the estimated CTE values at relatively lower temperatures show large scatter, or they are rather higher than the reported values for bulk Cu specimens in the range between 1.71 – 2.03 x $10^{-5}$ $K^{-1}$ [10]. However, the values at high temperature range are in quite good agreement with that for the bulk specimen. This suggests that the CTE of small samples can be estimated properly with the proposed technique. Similarly the CTE values for a small Au specimen during heating and cooling are shown in Fig.6. It was reported that the CTE of bulk Au is ranging between 1.42 – 1.67 x $10^{-5}$ $K^{-1}$ [10], which is also in agreement with the estimated CTE values for the Au specimen during cooling at the temperature range higher than 250 °C in the present study. The reason for the scattering of the measured values during heating is needed to be pursued.

In order to measure CTE with better accuracy, the problems caused by heating system such as the inhomogeneous heating are needed to be as small as possible. However, the heating and cooling rates of 50 and 300 K/min for Au and Cu specimens, respectively, for the present study are extremely higher than that for conventional techniques for the CTE measurement generally ranging between 1-10 K/min. Therefore, a rapid measurement is also an advantage of this method. It is caused by the small specimen size as well as small apparatus set-up, resulting in the good homogeneity of the temperature of measurement system. Vibration also affects on the fringe space measurement by waving the fringe patterns. To overcome these difficulties, it is needed to set high modulus, well-designed LSCM on a damping device.

In this study the proposed method is applied for CTE measurement of small specimens. On the other hand, this method can be applied to the materials having the orientation dependence of the CTE. The physical properties of materials which have low-symmetric crystal structures may show the orientation dependence. For the measurement of such properties using conventional methods, a number of large single crystal specimens are needed. However, the growth of the single crystal is generally very difficult and time consuming. The smaller the size of the crystal is, the easier the single crystal

growth becomes. The typical specimen size for the CTE measurement with the proposed method is as small as the typical size of a grain in polycrystalline samples produced by conventional melting and casting method for various metallic materials. That is to say, the proposed method is applicable to measure the orientation dependence of the CTE of micron-size single crystal samples prepared by combining SEM-EBSD for the measurement of crystallographic orientation of each grain and FIB system for micron-size specimen fabrication as illustrated in Fig.7 schematically.

Also the method can be applied widely to measure the change in height or volume of a small specimen. For example, change in volume during the phase transition caused by the change in temperature, pressure or magnetic field, etc. The volume change caused by the change in gaseous partial pressure is also within the scope of the method. One may expect to apply this method for the evaluation of volume change of hydrogen absorbing materials as a function of hydrogen pressure and temperature, as well as the volume change of organic materials during drying.

## CONCLUDING REMARKS

A brief sketch of a novel method for the measurement of the coefficient of the thermal expansion (CTE) of specimens as small as several micron meters is depicted. This method needs no information on the specimen size and specimen location. There are no limitation on the shape and surface preparation for the measurement. Although, for an accurate measurement, several problems such as the change of the location of supporting bed during heating and cooling and the vibration are needed to be solved, it is shown that the CTE of Cu and Au small specimens can be measured under much higher heating rate.

It is also shown that the method can be applied for the measurement of orientation dependence of the CTE, and the volume change of specimens under various conditions.

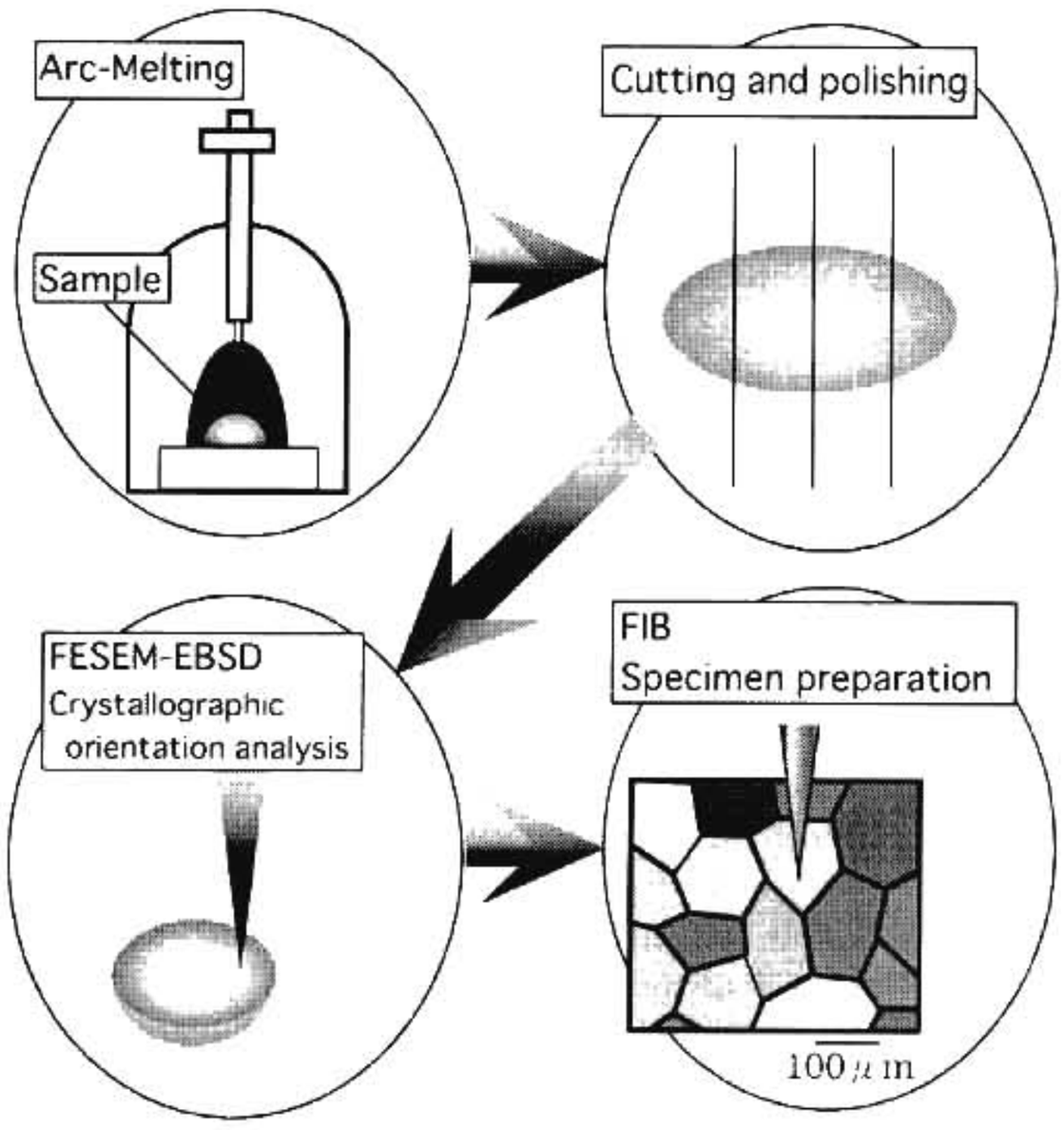



Fig.7 A scheme of the specimen preparation for the measurement of the orientation dependence of the CTE.

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