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On imaging metal grains at high temperature: Laser light scattering microscopy

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A simple device has been developed to meet the need of a base that is free from thermal expansion and is required in certain hot-stage microscopy studies. Further, images of sub-millimeter sized metal grains at high temperatures are noticed to under go vibration-like motion. In order to achieve stabilization of images, a unit called beam path cooler has been devised and its effect is examined.

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In surface analysis and related areas, some interesting applications of laser light scattering has been noticed in the past and current research[1-10]. Deep defocus laser light scattering microscopy investigations were carried out[11] on sub-millimeter sized grains of Cu, Ag and Zn in the temperature range 20-70°C; later the study was extended to a wider temperature range 20-400°C. The high temperatures necessitated large working distance objective (LWDO) water-cooled microscope[12] (working distance d_w = forward focal length of objective lens f_{fo} = 80 mm; aperture Φ = 3 mm) and sample stage, which must not exhibit any thermal expansion, at least in the direction of interest. We designed and fabricated such a stage with mild steel[13] and called it V$^+$ZET (table with zero expansion in vertical up-ward direction). During the experiments, it was noticed that the images of Au metal grains at high temperatures exhibited vibration-like motion. In order to achieve stabilization of images, a unit called beam path cooler (BPC) was devised and its effect was examined. The aim of this paper is to report the details and performance evaluation of the V$^+$ZET and BPC developed in this laboratory.

Experimental Procedure

Sample and sample holder

Sub-millimeter sized irregular shaped grains of gold (Sigma-Aldrich make; 99.999% purity) were employed in the present study. Because of the high surface reflectance and resistance to oxidation, Au was chosen for initial studies. The reflection coefficients of Au and carbon black are 0.75 and 0.003 respectively, in the visible spectrum[14]. Amorphous powder of MnO_2 (whose surface reflection coefficient may probably be close to that of carbon black) was used to suppress the back-ground scattering. A fused silica optical flat (0.8 mm thick and 8 mm diameter) was used as sample holder, in view of its extremely low thermal expansion. Coefficients of thermal expansion[15] of fused silica and gold are 0.4×10^{-6} K^{-1} and 14×10^{-6} K^{-1} respectively.

Assembly of V$^+$ZET

The V$^+$ZET assembly is shown in Fig. 1a. A heavy mild steel metal block (200×80×20 mm^3) was cut into the shape shown in Fig. 1b and a 400 W flat heater was coupled to it; electric current was fed to the heater via a servo voltage stabilizer and auto-transformer. The block was rested on a pair of 150 mm long metal pipes. The distance between these two pipes was maintained by another set of pipes. Thermostatic water was pumped continuously through the pipe system, so that changes in the dimensions of the supports (due to variations in temperature) were eliminated. Thin, identical mica sheets acted as thermal barriers between hot and cold surfaces (of the block and its supports).

A plane AB passing through V$^+$ZET is defined such that the body rests on supports s_1 and s_2 along plane AB (Fig. 1b). When the V$^+$ZET is heated with the help of heater H, the body defined by plane AB cannot expand down-wards, because of the constraints $s_1(b_1)$, $s_2(b_2)$. As such the possibility is that it expands only above the plane AB. Since the plane C (called

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critical plane) lies in plane AB, it should experience no expansion (either up-wards or down-wards) because it is identical to the base plane[13]. Therefore, it is expected that the central point of the metal block should be free from thermal expansion in any direction. Whereas entire upper surface (area = 50 × 40 mm^2) of the trough should be free from thermal expansion in positive vertical direction (hence the name V$^+$ZET). In order to isolate V$^+$ZET from vibrations, the set-up was positioned on a 35×35×35 cm heavy duty base. Thick sheet of rubber was placed between the base and concrete flooring.

The performance of V$^+$ZET was evaluated with the help of a traveling microscope and thermocouple based temperature sensors, by making several observational runs on each surface (temperature range of 20-200°C); the expansion profiles are presented in Figs 2a and 2b. It may be of interest to note that curves f and j (Fig. 2b) were due to measurements made on two points f and j, present on the same plane (at two different positions). Yet, they showed different profiles (levels of thermal expansion) and this observation may need an explanation. It may be noted that the point f and j are close to and far way from the plane C respectively. Interestingly, the point f, which is close to plane C, exhibits only small expansion and the point j, considerably away from plane C, exhibits relatively large expansion. Clearly, the variation in expansion, measured from plane C is a smooth change, from a low value to high value. Since the effective length of an element between plane C and a point x increases, as the point x is moved down-wards. If the distance measured between plane C and point x is *L*, and the expansion produced is Δ*L*, then simply Δ*L* is proportional to *L*. As such point J shows more expansion than point f.

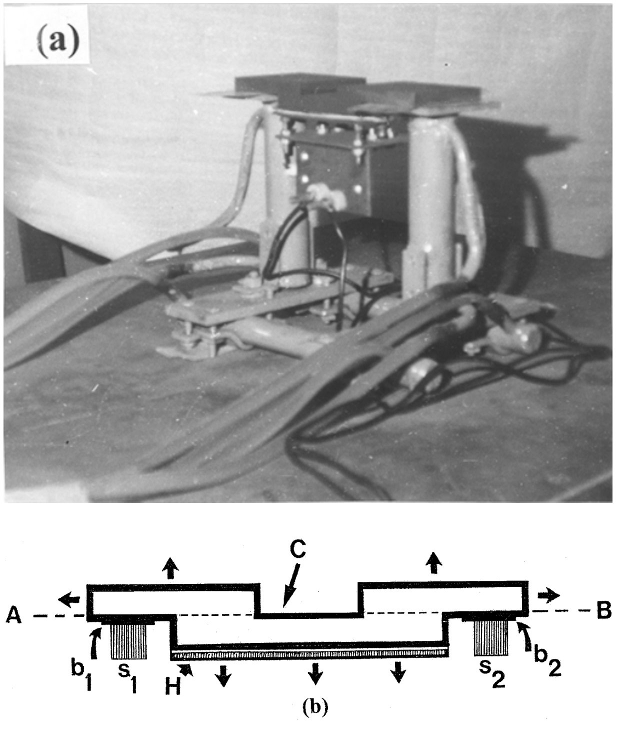

Fig.1—(a) Photograph of V$^+$ZET assembly: (**a**) flat-form, (**b**) expansion free zone, (**c**) heater, (**d**) vertical Supports, (**e**) water flow system, and (b) schematic representation of the stable plane AB and critical plane C and possible directions of expansion; b$_1$, b$_2$ are thermal barriers and s$_1$, s$_2$ are water-cooled supports.

Beam path cooler (BPC) and image quality

A 670 nm, 10 mW laser beam falls (at an oblique angle) on the experimental metal grain placed on the V$^+$ZET and undergoes scattering, while a part of it enters the microscope, after traveling a distance of 80 mm.

In order to prevent drift of the focal plane (caused by the thermal expansion of the body of microscope), an elaborate copper sheet made heat shield (fitted to a trolley that moves on a track) with circulating water was devised to absorb heat coming from V$^+$ZET. However, the experimental runs showed that image of the Au metal grain remained stable up to about 90°C

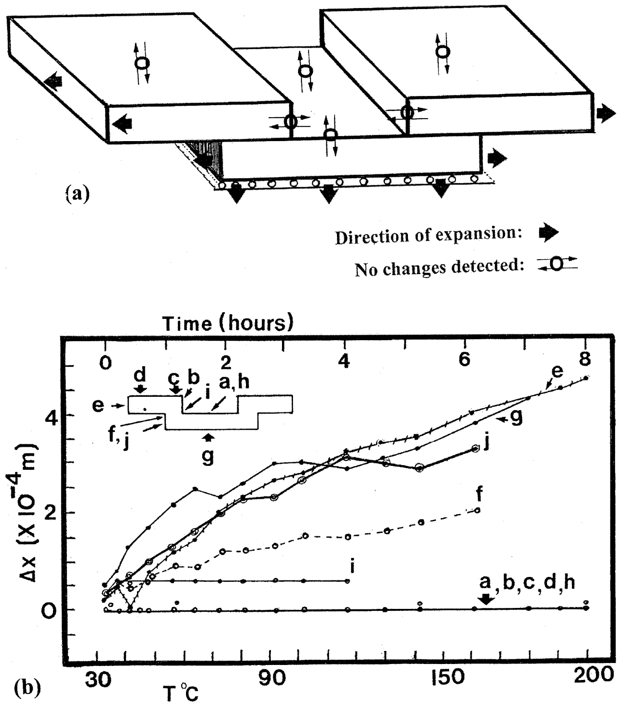

Fig.2—(a) Experimentally detected over-all expansion profile of V$^+$ZET. (b) The curves (a-j) indicate the expansion characteristics of different faces. The inset shows the points where the observations were made to generate the curves (a-j).

and then the image underwent vibration-like motion in x-y plane, coupled with fluctuating deformation. The oscillations became intense when temperature reached 200°C. Exposure time about 90 s was needed to record the image on 400 ASA B&W film. These oscillations thus blocked finer details in the images. This aspect is illustrated in Fig. 3.

Results and Discussion

It was understood that the air currents, promoted by the temperature gradients gave rise to fluctuations in refractive index of air, thus leading to vibration-like motion in the image. The effect was almost identical to the twinkling of starlight in the night sky. The three possible ways to eliminate the fluctuations in the refractive index were: (i) to allow the light to pass through vacuum, (ii) to cool the path of the light beam and (iii) to adopt computer aided image restoration. The last technique (iii) was not within our reach. With respect to the technique (i), heating of sub-millimeter sized grains of metals and alloys in vacuum might trigger evaporation. Due to such evaporation, the surface may under go continuous micro-topographical modifications, which in turn can strongly influence the laser light scattering profiles (LLSP). As a result, the detected LLSP shall be a sum of the laser light scattering due to the thermally triggered changes in the sub-millimeter sized metal grains and the vacuum induced continuous changes in the surface of a given grain. It may almost be impossible to separate the two quantities, and the aim of the study would become unattainable. The level of surface evaporation may be different for different metals and alloys. However, since metals and alloys with both high and low melting temperatures are planned to be investigated, by heating them to high temperatures, for prolonged time (ranging from 4-20 h), it is desirable to avoid heating in vacuum. Therefore, the second option was chosen.

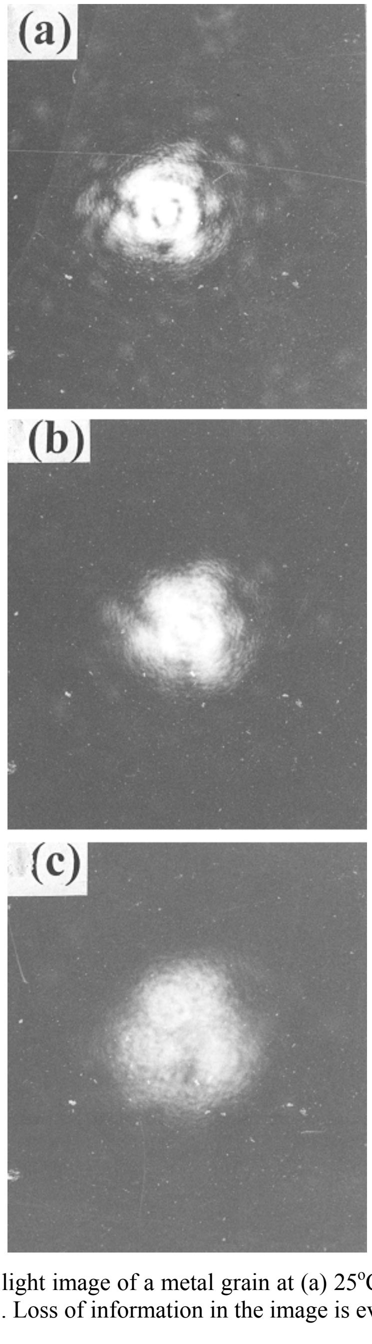

Fig.3—Laser light image of a metal grain at (a) 25°C, (b) 105.6°C and (c) 200°C. Loss of information in the image is evident.

A beam path cooler (BPC) has been designed and developed employing water circulation systems. The large working distance objective (LWDO) microscope with V^+ZET, heat shield and BPC systems is shown in Fig. 4. A fused quartz optical flat was used to prevent the entry of hot air into cool zone of the beam path. The BPC worked effectively. Fig. 5 shows the improvement in the quality of image recorded at 200°C. It may be noted that there is no distortion in the image, when compared with the Figs 3b and 3c. The diffusiveness of the image (Fig. 5c) was due to intrinsic changes[16] that took place in the grain at high temperature. The only disadvantage noticed was that, due to the reflection of light by optical flat, the intensity of image reduced considerably. With BPC in the active mode, the required exposure time was about 540 s (still the image was quite good). Fig. 5c is the laser light scattering microscopic image of a sub-millimeter sized metal (Au) grain at 200°C.

It may be noted that, if 90s of exposure time were employed in the case, where BPC was in the active mode, then no image could be recorded due to acute under exposure of film. And if 540 s of exposure time were to be used, when BPC was in the inactive (withdrawn) mode, then the film would be extremely overexposed and no details would be recorded. As such, recording the images with and without BPC in the active mode, shall not be possible with identical exposure times. Further, it may also be noted that if vibrations were still present (even after the induction of BPC), then that would be explicitly manifested in the images, in view of long exposure time (540 s). Therefore, the utility and action of BPC may be placed beyond any doubt.

Further, when the axis of the BPC and the optic axis of microscope coincided with each other, then the image was centrally located in the field of eye-lens. This condition is labeled as central position of BPC. Under such conditions, image of grain with and with out BPC in active mode were noticed to be identical. It was also noticed that the stabilized image experienced deformation, when BPC was shifted to either side from its central position. The reason for such deformation is as follows: A 75 mm long and 5 mm diameter tunnel defines the cool zone of BPC. If BPC is slightly moved sideways, then the outer envelop of the beam traveling through it shall come into contact with the tunnel surface. In all probability, that part of the beam (in contact with tunnel surface and thus blocked) shall not be able to participate in image formation, leading to loss of optical information, causing deformation of the image. This observation seems to be in close agreement with the

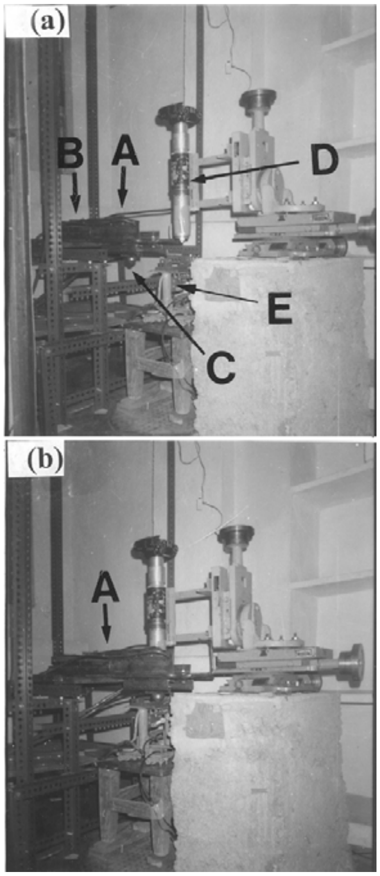

Fig.4—(a) The trolley (A) was in withdrawn state. (B): heat shield, (C): BPC, (D): LWDO microscope and (E): V$^+$ZET; (b) Trolley was moved forward – heat shield and BPC in active mode.

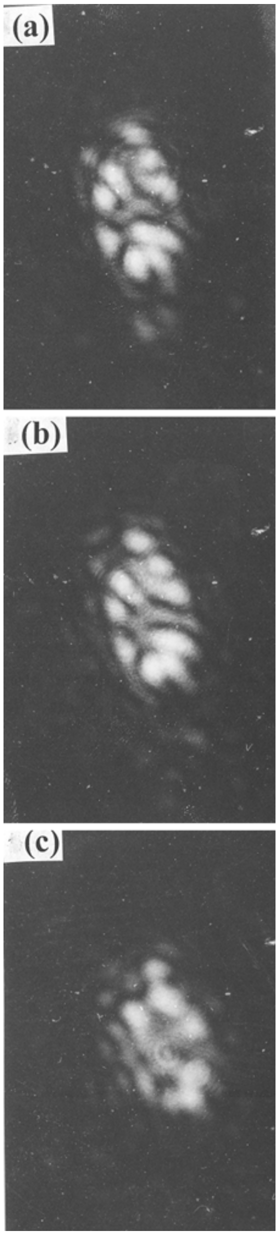

Fig.5—Images of a grain at (a) 33^{o}C, (b) 113.3^{o}C and (c) 200^{o}C; great improvement in the quality of image at high temperature is evident.

statement of Denisyuk[17] that, it is possible to synthesize an arbitrary image by controlling the wave field parameters.

Conclusions

The study establishes the functionality and utility of the V^+ZET-BPC system. It provides both thermal expansion free environment in +Z direction and also stabilized images at high temperature. As such the system can be highly useful in such studies that warrant expansion free hot stages. If mild steel be replaced by invar steel, then the expansions in lateral directions can be reduced drastically.

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