



Trade Science Inc.

# Materials Science

An Indian Journal

Short Communication

MSAIJ, 6(1), 2010 [68-70]

## Variable temperature laser light scattering microscopy studies on 10 - 100 $\mu$ m size grains of gold, aluminum, zinc and titanium: Role of relaxation time in thermally triggered volume changes

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Received: 22<sup>nd</sup> January, 2010 ; Accepted: 1<sup>st</sup> February, 2010

### ABSTRACT

Laser light scattering microscopy investigations on Au, Al, Zn and Ti metal grains were made at different temperatures; a thermal volume-strain variation parameter was defined. A modified equation for thermal expansion that took relaxation time (time needed for complete expansion due to injection of heat) into consideration was given and constancy of a constant (b), used in the modified equation was established.

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### KEYWORDS

VTLLSM;  
Thermal volume-strain variation;  
Thermal expansion-relaxation time.

### INTRODUCTION

The thermal behavior of small metal grains in the size range of 10-100 $\mu$ m was investigated by employing variable temperature laser light scattering microscopy (VTLLSM), in a temperature range of 25 to 250°C, using a 670nm, 10mW diode laser. Experimental details and interpretation of images were reported earlier<sup>[1,2]</sup>. The relation between thermally triggered volume changes and role of relaxation time is discussed in the present report.

### OBSERVATIONS AND DISCUSSION

The area of bright patches (BPs) recorded in a heating run on a metal (Zn) grain are shown in Figure 1 as an example. The maximum and minimum total areas of BPs  $A_{\max}$  and  $A_{\min}$  were related by a factor  $\omega$ , which may be defined<sup>[3]</sup> as

$$\omega = (A_{\max} - A_{\min}) / (A_{\max} + A_{\min}) \quad (1)$$

The factor  $\omega$  represents thermal volume-strain variation. The  $\omega$  values of title metals obtained at different rates of heating (dT/dt) are shown in Figure 2. All the curves showed initial brief increase and then continuous decrease. Continuous decrease in the average value  $\langle \omega \rangle$  is interpreted as due to restricted response to the imposed thermal field, caused by insufficient time for expansion to fullest extent, permitted by the temperature difference. In order to explain the observations, the classical equation for thermal expansion<sup>[4]</sup> is modified as,

$$V_T = V_0 \{1 + 3\alpha \Delta T / \exp\{t^{-1}[1 - b^{(t-t_0)}]\}\} \quad (2)$$

Where  $V_0$  and  $V_T$  are initial and final volumes at temperatures  $T_0$  and  $T$  respectively;  $\Delta T = T - T_0$ . The coefficient of thermal (linear) expansion is represented by  $\alpha$ . (Even though  $\alpha$  varies with temperature, a constant value of  $\alpha$  is assumed in the equation, since the varia-

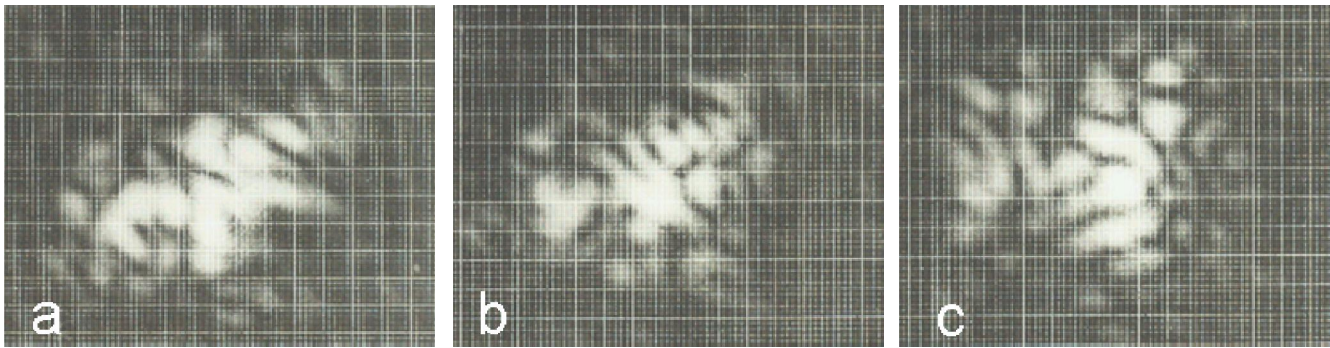


Figure 1 : VTLLS microscopy images of a grain of zinc at (a) 30, (b) 120, (c) 200°C;  $dT/dt = 0.2^\circ/\text{min}$ .

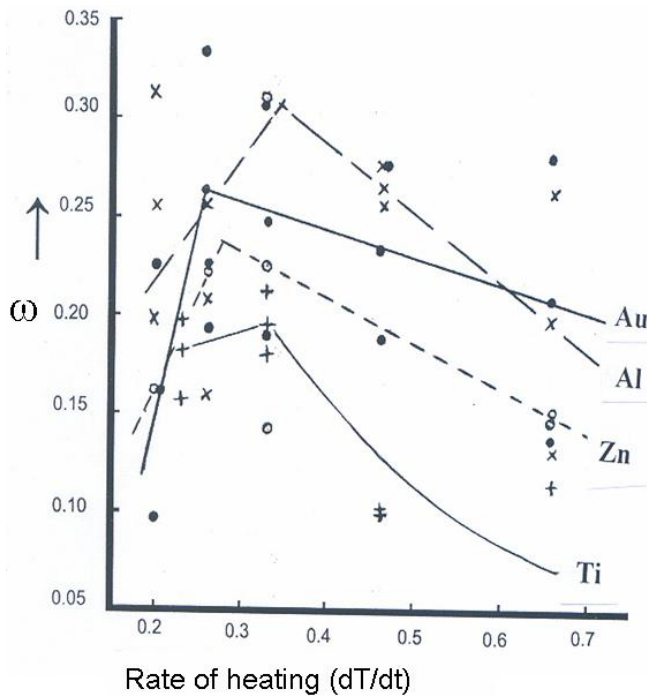


Figure 2 :  $\omega$  vs.  $dT/dt$  curves of (a) gold, (b) aluminum, (c) zinc and (d) titanium grains. The peaks correspond to critical relaxation time  $t_c$ .

tions are not too large in comparison with  $\alpha$  it self, as seen from TABLE 1).

Relaxation time and actual time (available in a given heat-run, determined by rate of heating) are indicated by  $t_c$  and  $t$  respectively;  $b$  is a constant. The Eq.2 gives the following results for different experimental conditions: (i) If  $t = 0$ , then  $V_T = V_o$ . It means that in the absence of time for expansion (or relaxation), there will be no expansion (even though  $\Delta T \neq 0$ ). (ii) If  $t_c = t$ , then Eq.2 reduces to the usual  $V_T = V_o \{1 + 3 \alpha \Delta T\}$ . (iii) When  $t > t_c$ , then complete expansion takes place. Therefore, notional time is infinite. Once again Eq.2 reduces to the usual form. (iv) If  $t < t_c$ , then only restricted expansion takes place, due to insufficient

time for complete relaxation. The shapes of the curves are interpreted as follows: During the low rates of heating, grains have sufficient time for complete expansion. As such  $\omega$  was increasing with increase in  $dT/dt$  (more expansion for more rapid increase in temperature). Such increase occurs only as long as the available time for expansion is larger than the (critical) relaxation time  $t_c$ . The peak noticed in each curve therefore may correspond to the condition,  $t = t_c$ . Once the available time  $t$  is lesser than the critical relaxation time  $t_c$ , grains fail to show complete response to an imposed temperature differential, since a new expansion requirement gets imposed on the grain, before it fulfills the earlier requirement. As such  $\omega$  vs.  $dT/dt$  curves show a constant decline. It is known<sup>[5,6]</sup> in the field of first order phase transitions that in case of high heating rates, the materials melt, with out going through the expected transitions. The present situation may be akin to it (in the sense that both processes need some minimum time period to, accomplish the warranted adjustments on atomic and molecular scale). It is known that  $\alpha$  varies with temperature<sup>[7]</sup>. Therefore average values of  $\alpha$  (estimated from the graphs<sup>[7]</sup>) for the relevant temperature ranges were used in estimation of constant  $b$  of Eq.2. The parameters derived from the experimental conditions, yielding the peak values, are shown in TABLE 1.

TABLE 1 : The  $t_c$  and  $\langle b \rangle$  values for four metals

Metal	$\alpha^{[12]}$	Estimated $\alpha$ values <sup>[7]</sup>	$t_c$ (*)	$\langle b \rangle$
Ti	$8.4 \times 10^{-6}$	$9.37 \times 10^{-6}$	182	1.0754
Au	$14.2 \times 10^{-6}$	$14.04 \times 10^{-6}$	231	0.9648
Al	$23.6 \times 10^{-6}$	$25.86 \times 10^{-6}$	172	0.9963
Zn	$27 \times 10^{-6}$	$15.78 \times 10^{-6}$ ( $\perp c$ -axis)	218	1.1814

(\*)  $t_c$  is measured as time (in seconds) needed for  $1^\circ\text{C}$  temperature rise.

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The reasonably constant value of  $b$  may be indicative of the validity of Eq.2. It seems that the differential expansion  $V_E - V_T$  (where  $V_E$  and  $V_T$  are the experimentally detected volume at a temperature  $T$ , and the volume expected at that temperature respectively) of a grain, becomes more and more dominant<sup>[3]</sup>, as the grain size is reduced, paving way for the manifestation of role of defects<sup>[8-11]</sup>. The VTLLSM studies on this aspect shall be discussed separately.

### ACKNOWLEDGEMENTS

The authors sincerely thank Defense Research Development Organization, Ministry of Defense, Government of India for funding a project on laser light scattering by metal grains, under which the studies reported here were carried out. Authors are also thankful to Prof. P.B.V. Prasad (deceased), who actually lead the project and completed it successfully.

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