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# Damping Associated with Incipient Melting in Aluminum-Indium Alloys

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The strain amplitude dependent damping of binary aluminum-indium alloys containing nominally 0.6 to 17.3 weight percent indium was studied. A dynamic mechanical analyzer was used to measure the damping capacity of these materials. Pure aluminum (99.99%) exhibited strain dependent damping at strain values as low as 70 $\mu\varepsilon$. The addition of 0.6 weight percent indium reduced the strain independent damping by a factor of 2, but the strain dependent damping was equivalent to that of pure aluminum. Binary aluminum-indium alloys containing 4, 8, 12, and 16 weight percent indium exhibited a general increase in loss factor with increasing indium content; however, the strain dependent damping was no greater than that of the pure aluminum sample. No significant increase in damping was observed when the binary alloys were tested at temperatures above the melting point of indium. Two damping peaks were observed near the eutectic melting point when tested at 10 Hz and differential scanning calorimetry verified both of these peaks as due to the melting of the indium inclusions. It was concluded that the higher temperature damping peak was associated with smaller indium inclusions and that the damping peaks were related to the solute segregation associated with the binary eutectic reaction.

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## ABSTRACT

*The strain amplitude dependent damping of binary aluminum-indium alloys containing nominally 0.6 to 17.3 weight percent indium was studied. A dynamic mechanical analyzer was used to measure the damping capacity of these materials. Pure aluminum (99.99%) exhibited strain dependent damping at strain values as low as 70 microstrain. The addition of 0.6 weight percent indium reduced the strain independent damping by a factor of 2, but the strain dependent damping was equivalent to that of pure aluminum. Binary aluminum-indium alloys containing 4, 8, 12, and 16 weight percent indium exhibited a general increase in loss factor with increasing indium content; however, the strain dependent damping was no greater than that of the pure aluminum sample. No significant increase in damping was observed when the binary alloys were tested at temperatures above the melting point of indium. Two damping peaks were observed near the eutectic melting point when tested at 10 Hz, and differential scanning calorimetry verified both of these peaks as due to the melting of the indium inclusions. It was concluded that the higher temperature damping peak was associated with smaller indium inclusions and that the damping peaks were related to the solute segregation associated with the binary eutectic reaction.*

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## INTRODUCTION

The typical structural aluminum (Al) has a high stiffness but a low specific damping capacity. The typical loss factor for a precipitation hardened aluminum alloy is between $10^{-3}$ and $10^{-4}$. Metal matrix composites have shown increased damping[1] but these materials cannot be considered high damping because they have loss factors less than $10^{-2}$.[2] An alternate approach to the development of a high damping composite would be by the incorporation of a viscoelastic fiber in addition to the stiff fibers used for reinforcement. Thus, the matrix would provide the structural stiffness and the various fibers would provide the desired damping capacity and the added stiffness. Although the composite material would show a lower stiffness than the stiff metal matrix composite, the increase in damping capacity may be of greater importance.

Indium (In) is a viscoelastic metal with an ultimate tensile strength of 3.1 MPa (450 psi) at room temperature. The loss factor of indium has been reported to vary from 0.06 at room temperature to 0.2 at 100°C (212°F).[3] The melting point of pure indium is 156°C (313°F) and when combined with aluminum forms and immiscible alloy system, as shown in the phase diagram in Fig. 1. An alloy of 17.3 weight percent indium will solidify by a monotectic reaction ($L \rightarrow Al+L_2$) which produces a continuous aluminum matrix with an indium rich ($L_2$) entrapped liquid. At 156°C the indium rich liquid will solidify by the eutectic reaction $L_2 \rightarrow Al+In$. This final eutectic reaction will normally produce inclusions which are single-phase, i.e., pure indium. The small weight fraction of aluminum produced during the eutectic reaction is "divorced" to the preexisting aluminum matrix. Thus the resulting microstructure will consist of an aluminum matrix with a dispersion of indium inclusions.

The objective of this investigation was to examine the effect of a viscoelastic inclusion, indium, on the damping capacity of aluminum. It was expected that the composite microstructure would demonstrate strain dependent damping as a result of microplasticity (dislocation motion) within the inclusions. In addition, high temperature loss factor measurements were used to determine the damping associated with liquid inclusions. It was also expected that the first order transformation (the eutectic reaction) at 156°C would produce both an anomalous modulus effect and a frequency dependent loss peak. The relaxation time of the eutectic reaction determines the frequency at which peak damping is observed. The following formula from Nowick and Berry[4] describes the relaxation time, $\tau$, as a function of the radius, $r$, of the second phase particle for a two phase material during a first order transformation.

$$\tau = \frac{r^2}{3\ D\ Vf} \tag{1}$$

where $D$ is the diffusivity and $Vf$ is the volume fraction of the second phase particle. It should be noted that the relaxation time will be strongly dependent upon the size of the second phase, such that smaller particles exhibit shorter relaxation times.

## EXPERIMENTAL PROCEDURE

Binary aluminum-indium alloys, with nominal compositions of 0.6, 4, 6, 8, 12, and 16 weight percent indium, were arc-melted in an argon gas atmosphere. These alloys were prepared from indium and aluminum metals which each had a metallic purity better than 99.99%. The total weight of each arc-melted button was below 10 g to assure a homogeneous melt. The Al-17% In alloy was produced by induction melting in an argon gas atmosphere and was solidified at a slow rate using a ceramic insulator to produce a coarse distribution of indium particles. Each sample was then cold rolled 30%, annealed at a temperature of 532°C (990°F), then cold-rolled and annealed again to produce a nominal sample thickness of 1 mm. Rectangular beam coupons were cut from the rolled slab, using a diamond saw, to produce sample shapes with nominal dimensions 40 mm x 10 mm x 1 mm.

A DuPont dynamic mechanical analyzer model 983 (DMA) was used to measure the damping response of the test coupons. At room temperature and a fixed frequency of 0.1 Hz, the strain amplitude was varied from 20 to 300 microstrain ($\mu\varepsilon$) by changing the oscillation amplitude and clamping distance between the pivot arms, Fig. 2. The driver arm produces a sinusoidal displacement inducing both a shear and bending stress. The damping capacity was measured as the loss factor which is equal to the tangent of the phase angle, tan δ, between the stress and the strain. Elevated temperature tests, from 100-200°C (212-392°F) were conducted at a strain amplitude of 70 με at both 1 and 10 Hz. A heating ramp of 1°C (3.6°F) per minute and a helium gas atmosphere was used to minimize the temperature lag of the sample with respect to the furnace-controlling thermocouple.

The eutectic melting temperature of the binary alloys was established using a Perkin-Elmer differential scanning calorimeter model 7. Metallographic samples were prepared by mechanical polishing and etching in a hot aqueous solution of NaOH. Cross-sectional samples were cut to view the long transverse microstructures of the binary alloys. Electron microscopy studies were performed at The University of Michigan Electron Microbeam Analysis Laboratory. Thin foils for transmission electron microscopy were prepared by twin jet electropolishing in a solution of 20% nitric acid (by volume) and methanol.

## RESULTS

The results of the room temperature damping capacity measurements for the pure aluminum and binary aluminum-indium alloys are shown in Figs. 3 and 4 and their microstructures are shown in Fig. 5. Each alloy exhibits a transition to strain amplitude dependent damping at approximately 70 με. A comparison between the aluminum and the binary Al-0.6% In alloy is shown in Fig. 3. The addition of 0.6 indium reduced the strain independent damping by a factor of 2, but the strain dependent damping was equivalent to that of the pure aluminum. The strain independent damping of the pure aluminum was also greater than the binary aluminum-indium alloys, with the exception of the two highest indium concentrations, Al-12% In and Al-16% In. In general, the damping capacity of the aluminum-indium alloys and the size of the indium stringers increased with increasing indium content, as illustrated in Figs. 4 and 5.

The results of a typical 1 Hz temperature scan is shown in Fig. 6 for Al-6% In. A first order transformation was observed between 160 and 170°C (320 and 338°F). The change in the storage modulus with respect to temperature shows an anomalous behavior in this temperature range. The eutectic melting temperature of 156°C was verified by differential scanning calorimetry (DSC). However, The DSC results also revealed a second melting peak at 160°C, as shown in Fig. 7. Figure 8 shows that both melting peaks were observed with the DMA during a 10 Hz temperature scan. The loss factor associated with this transformation did not vary significantly with respect to increasing weight percentage of indium, as shown in Fig. 9. Although the total damping appears to increase with indium content, the difference between the peak height and the background is nearly constant.

At the eutectic melting temperature, the binary alloys exhibit strain dependent damping as demonstrated by the Al-17.3% In alloy in Fig. 10. This particular alloy had a coarse distribution of indium particles due to its slow cooling rate from the melt. The temperature scan in Fig. 11 shows a large damping peak to background ratio at lower frequencies for the Al-17.3% In alloy. When measuring the loss factor at the eutectic temperature for various oscillation frequencies, the relative height of the peak was observed to increase from values of 0.002 at 1 Hz to 0.014 at low frequencies of 0.1 Hz.

## DISCUSSION

The strain dependent damping of the aluminum-indium alloys appears to be associated with dislocation motion in both the aluminum matrix and the indium particles. Thus the damping of the aluminum-indium alloys increases with increasing indium content, but the total damping is less than that of the pure aluminum. This may indicate that the damping contribution from the matrix decreases with increasing indium content. This effect may be explained if we associate the magnitude of the matrix damping with a mean-free-path of the dislocation motion. Upon the addition of second phase particles, the mean-free-path of the dislocations will decrease in two ways: the indium particles will inhibit the grain size during annealing and the indium particles will act as dislocation traps. Both of these effects are a function of the volume fraction of the second phase. Therefore the damping contribution from the matrix would be expected to decrease as the volume fraction of second phase is increased. A minimum would then be expected for the aluminum-indium alloys since the damping contribution from the indium particles would increase with increasing volume fraction. This minimum is approximately at the Al-4% In composition.

The addition of indium also affects the strain independent damping of the aluminum matrix, as shown in Fig. 3. The addition of a very small amount of indium (0.6%) reduces the loss factor to one-half that measured for pure aluminum, but this effect appears to be related to processing history. Electron microscopy studies have just begun to examine the differences in structure which results from the addition of indium and the subsequent processing. For example, a second Al-0.6% In alloy was processed without annealing and the microstructure is shown in Fig. 12. The microstructure shows a fine sub-grain structure with indium particles on the sub-grain boundary. However, this particular alloy shows a much higher loss factor. The loss factor measured for this sample was constant, with $\tan \delta = 0.016$, up to a strain amplitude of 150 $\mu\varepsilon$. Before any conclusions can be made with regard to the strain independent regime, further microstructural studies must be conducted.

The damping peak observed between 160 and 170°C is believed to be related to the eutectic melting temperature observed at 156°C by the differential scanning calorimeter. The difference in temperature is a reflection of the thermal lag associated with the DuPont DMA. The pivot arms are made of stainless steel and are in direct contact with the sample. Thus, the pivot arms act as a thermal reservoir with respect to the sample. This effect was minimized by flowing helium gas through the furnace as the temperature was ramped. The thermal lag for the aluminum-indium samples varied between 4 and 10°C (7 to 18°F).

Equation 1 provides a means to calculate the test frequency at which peak damping would be observed for a two-phase microstructure going through a first order transformation. In the present case, the reaction is a eutectic where the indium alloys with the surrounding aluminum matrix to form a liquid. Self diffusion of indium in the liquid and in the solid state near the melting point is approximately $10^{-5}$ $cm^2/s$ and $10^{-9}$ $cm^2/s$, respectively.[5] If the typical diameter of the indium inclusion is taken as 2 $\mu m$ and a volume fraction of 0.02 is assumed, relaxation times of 170 and 0.017 seconds are expected, using the self diffusion rates for indium in the solid and liquid states, respectively. These relaxation times correspond to test frequencies of approximately 0.01 and 60 Hz. Resolution of the damping peak was obtained at a test frequency of 0.1 Hz, which would indicate an intermediate diffusivity. The diffusion rate of aluminum in indium would be expected to be higher than the self diffusion of indium in the solid state since the atomic radius of aluminum is smaller than that of indium. Thus a diffusivity between $10^{-7}$ and $10^{-8}$ $cm^2/s$ may be reasonable. In terms of order of magnitude calculations, this would produce a relaxation time on the order of 10 seconds or a test frequency of 0.1 Hz. The peak observed at the higher test frequencies may then be related to a smaller indium particle. It should be noted that the melting temperature of indium is size dependent.[6] This effect is easily demonstrated by differential scanning calorimetry of an arc-melted Al-12% In alloy, as shown in Fig. 13. The moderate solidification rate will produce a fine structure of indium particles which melt at a higher temperature. Upon cold-working and subsequent annealing, the number of high melting indium particles is reduced, as observed in the DSC resulting in Fig. 7.

## CONCLUSIONS

The addition of indium to aluminum exhibited a general increase in loss factor with increasing indium content; however, the strain dependent damping was no greater than that of the pure aluminum sample. A precipitation hardening alloy would be more appropriate for evaluating the damping contribution resulting from the addition of a viscoelastic inclusion. No significant increase in damping was associated with liquid metal inclusions, but a large damping peak was observed which was associated with the eutectic transformation and the diffusion of aluminum solute in the indium inclusions.

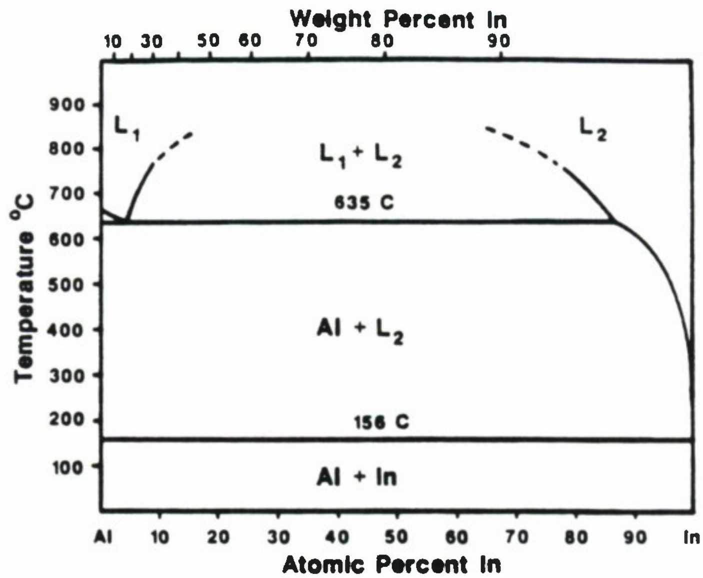


Fig . 1. Phase diagram of the Al-In binary system showing a liquid immiscibility gap.

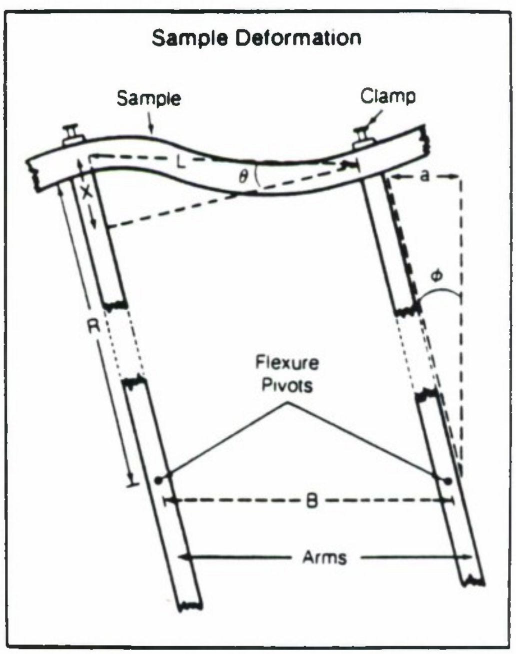


Fig. 2. Schematic representation of the pivot arm system for the DuPont DMA 983.

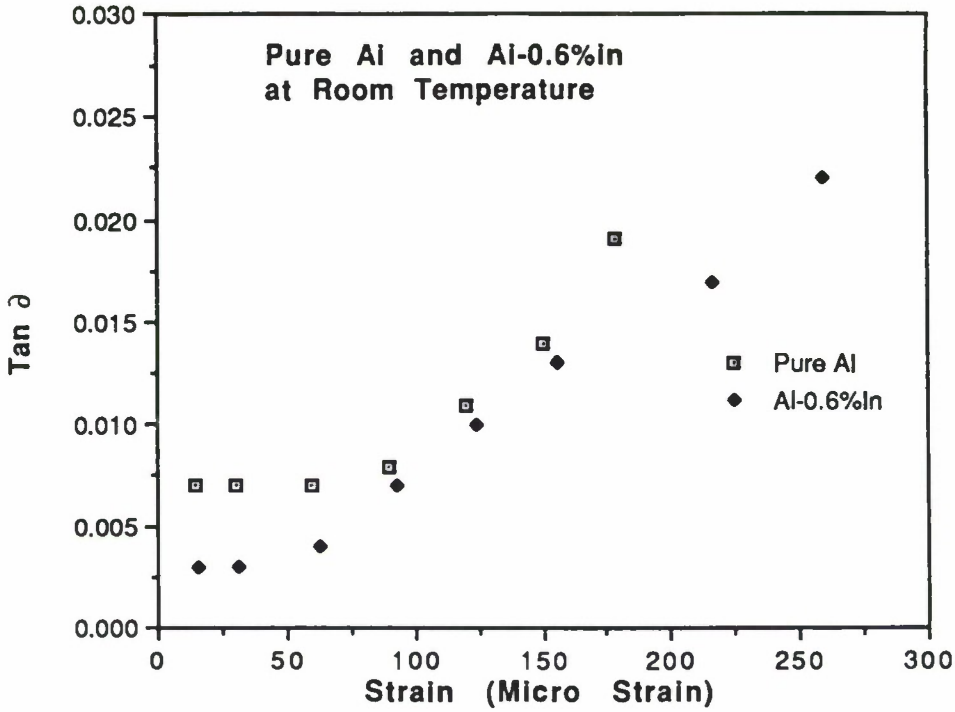


Fig. 3. Room temperature damping results of pure aluminum (99.99%) and Al-0.6 In for various strain amplitudes.

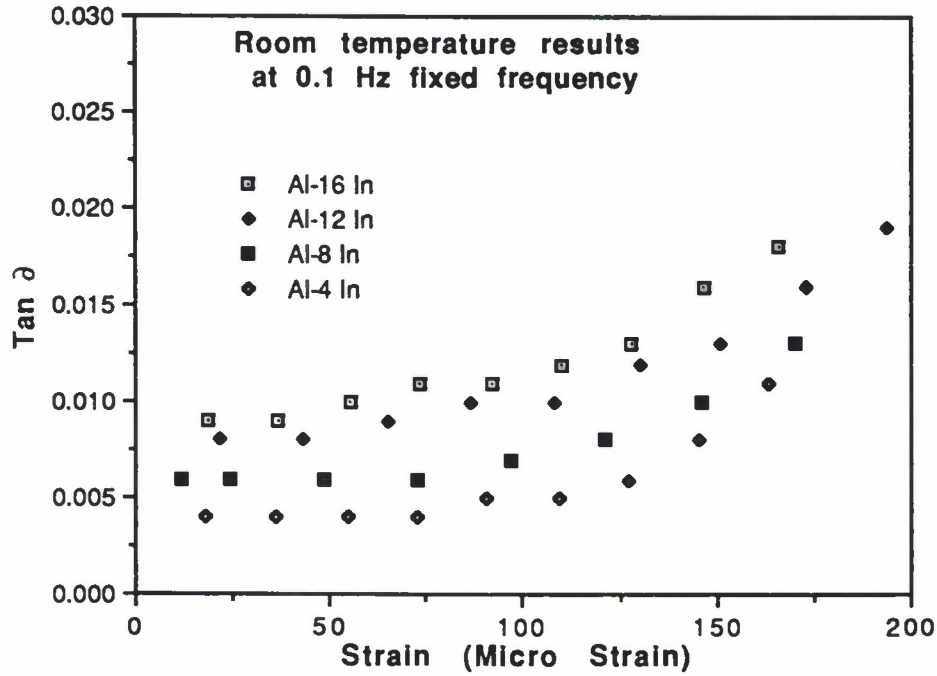


Fig. 4. Room temperature damping results of Al-4 In, Al-8 In, Al-12 In, and Al-16 In for several strain amplitudes.

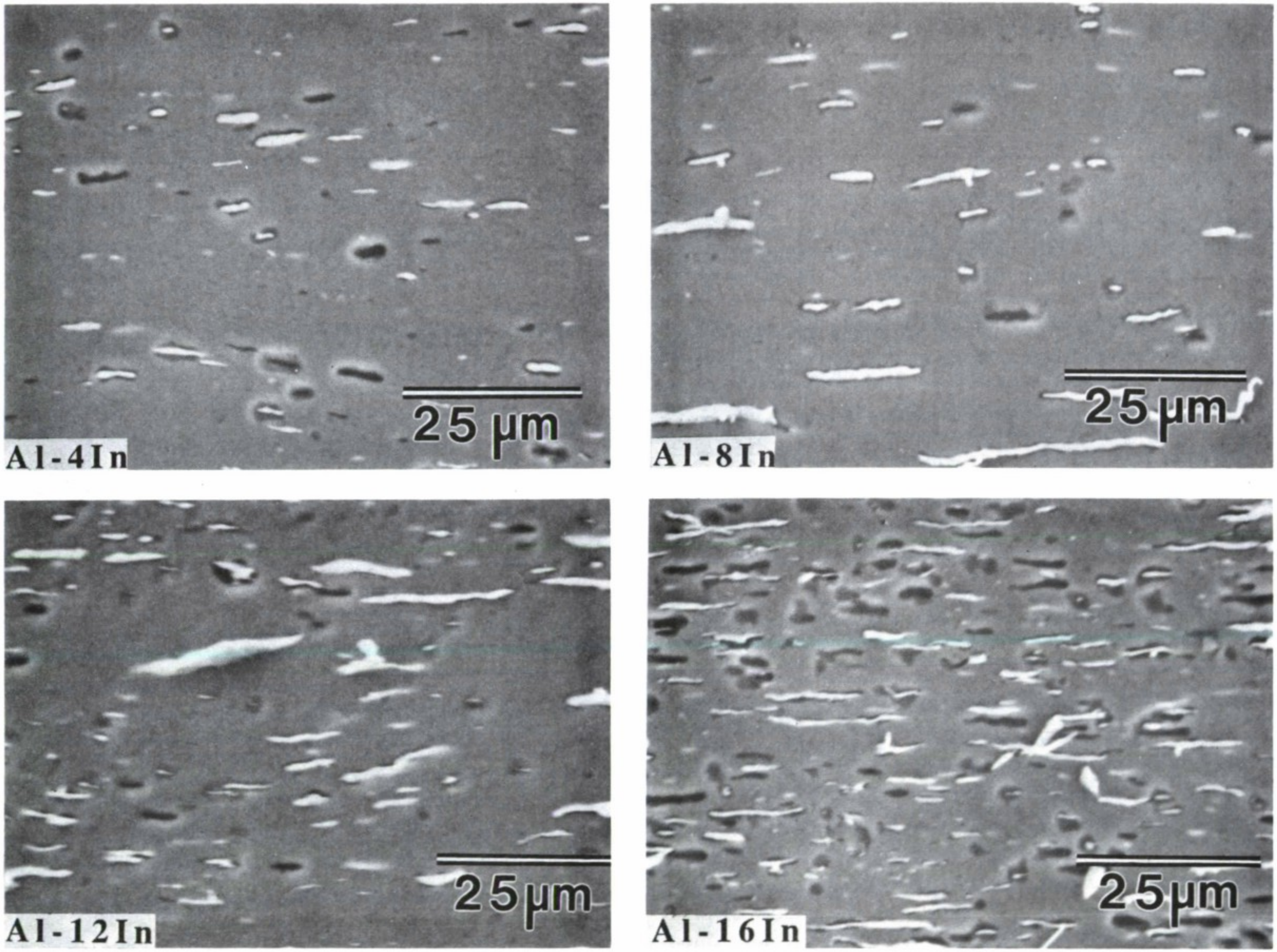


Fig. 5. Scanning electron micrographs of the Al-4 In, Al-8 In, Al-12 In, and Al-16 In alloys showing elongated indium stringers parallel to the rolling direction. The samples were polished and then etched in a warm aqueous solution of NaOh.

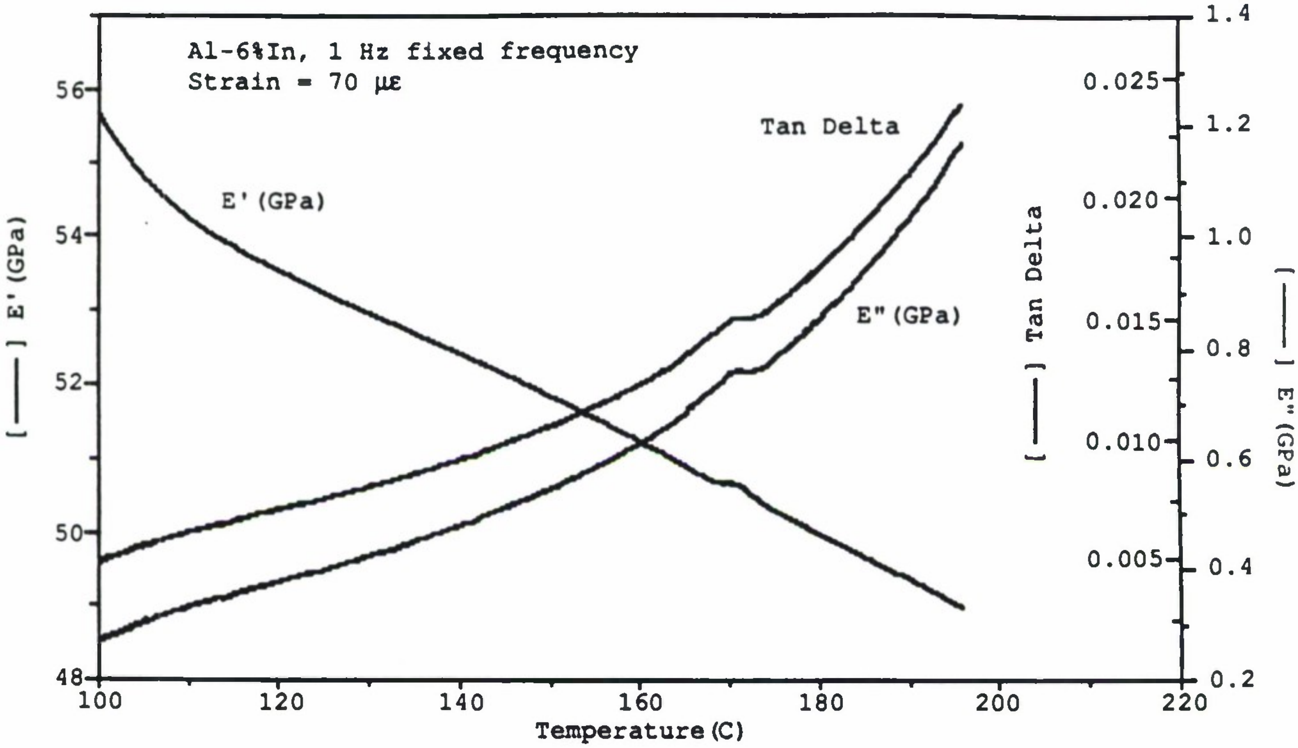


Fig. 6. Damping results from 100-200° for the Al-6 In alloy using a fixed frequency of 1 Hz and a strain amplitude of 70 $\mu$e.

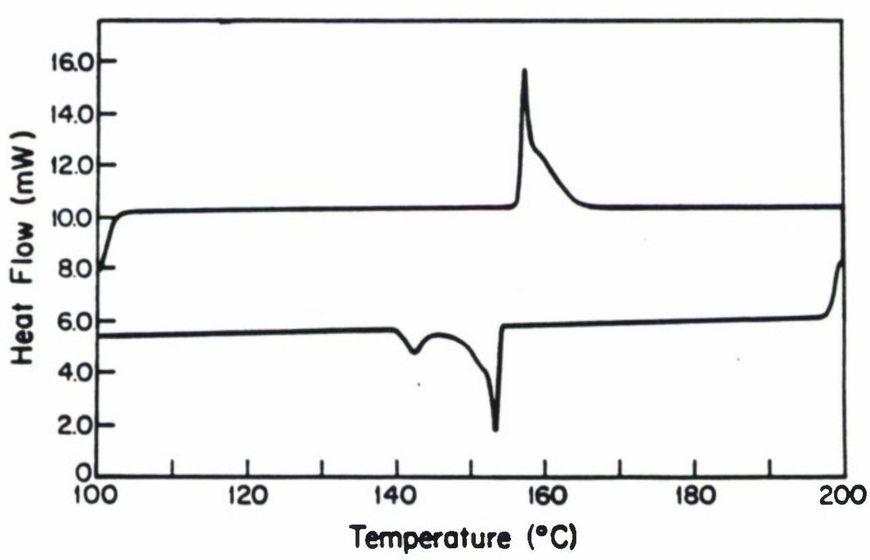


Fig. 7. Differential scanning calorimetry results from the Al-17.3 In alloy exhibiting two melting peaks upon heating (top line) and three solidification peaks upon cooling (bottom line). The smallest peak (140°C) is associated with solid nucleation of the finest indium particles.

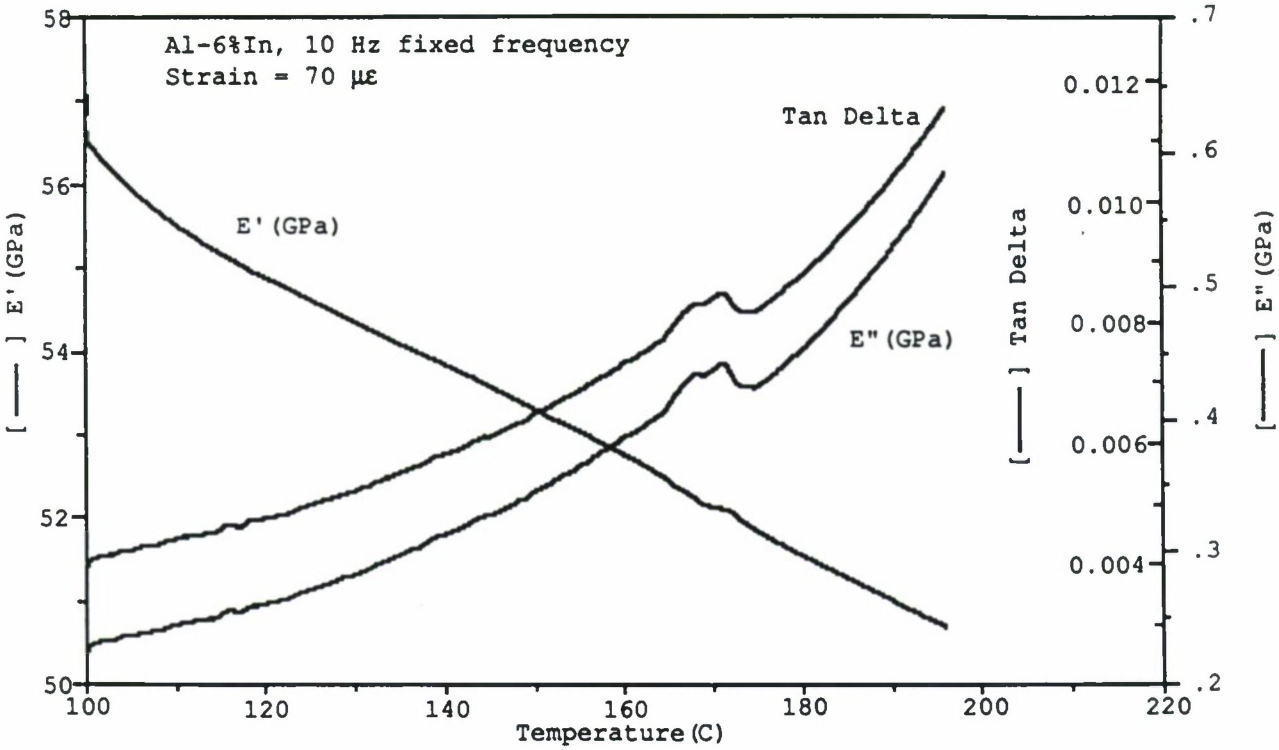


Fig. 8. Damping results for the Al-6 In alloy at 10 Hz fixed frequency and strain amplitude of 70 με showing two distinct damping peaks.

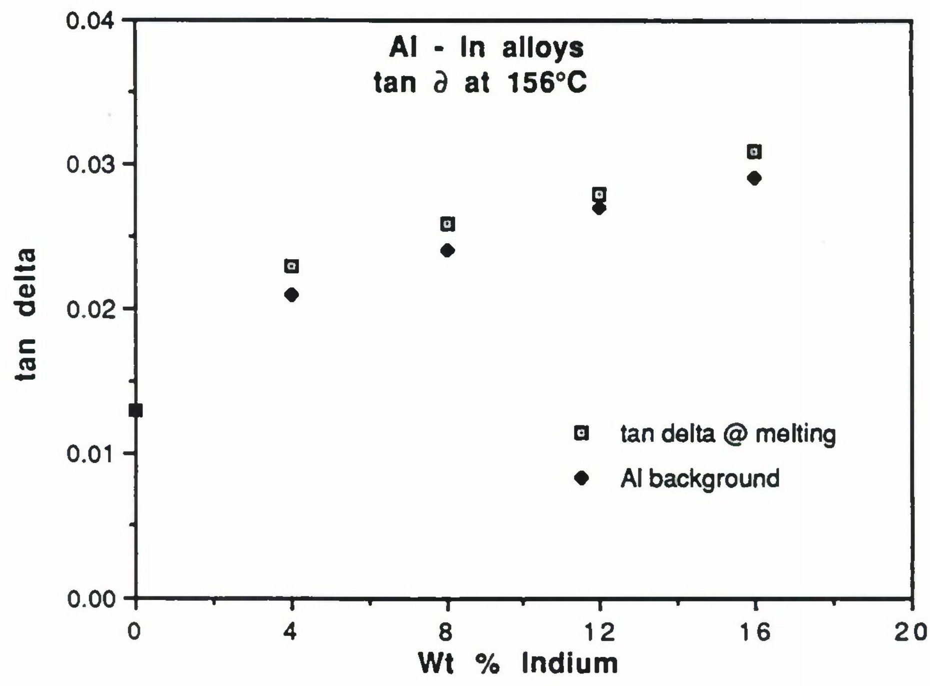


Fig. 9. DMA results indicating a slight increase of damping capacity at 156°C with increasing indium content and also showing damping contribution from the Al matrix.

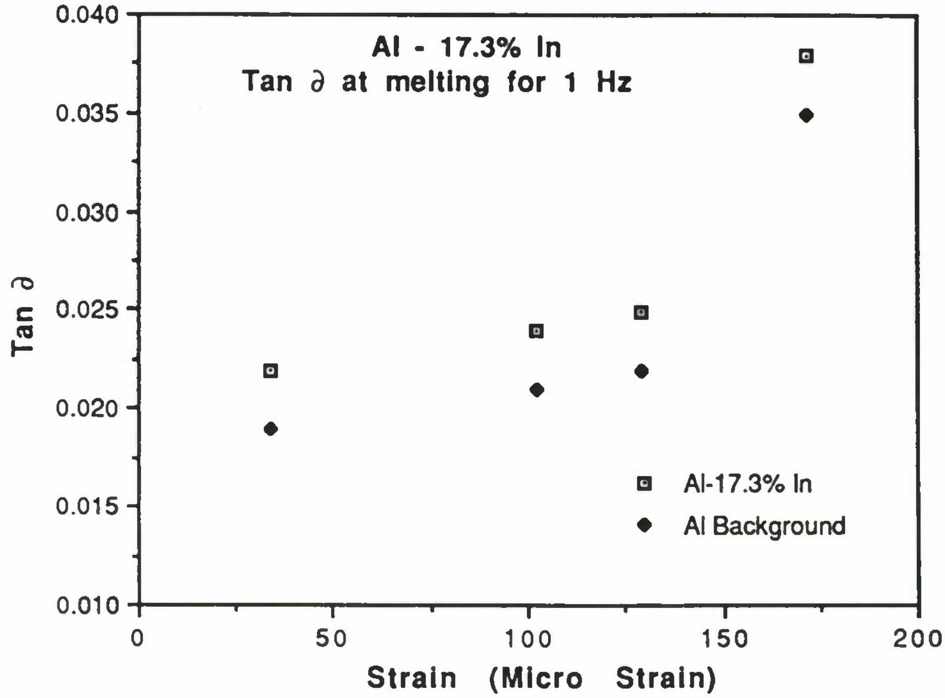


Fig. 10. Damping capacity results for the Al-17.3 In alloy at 156°C at different strain amplitudes.

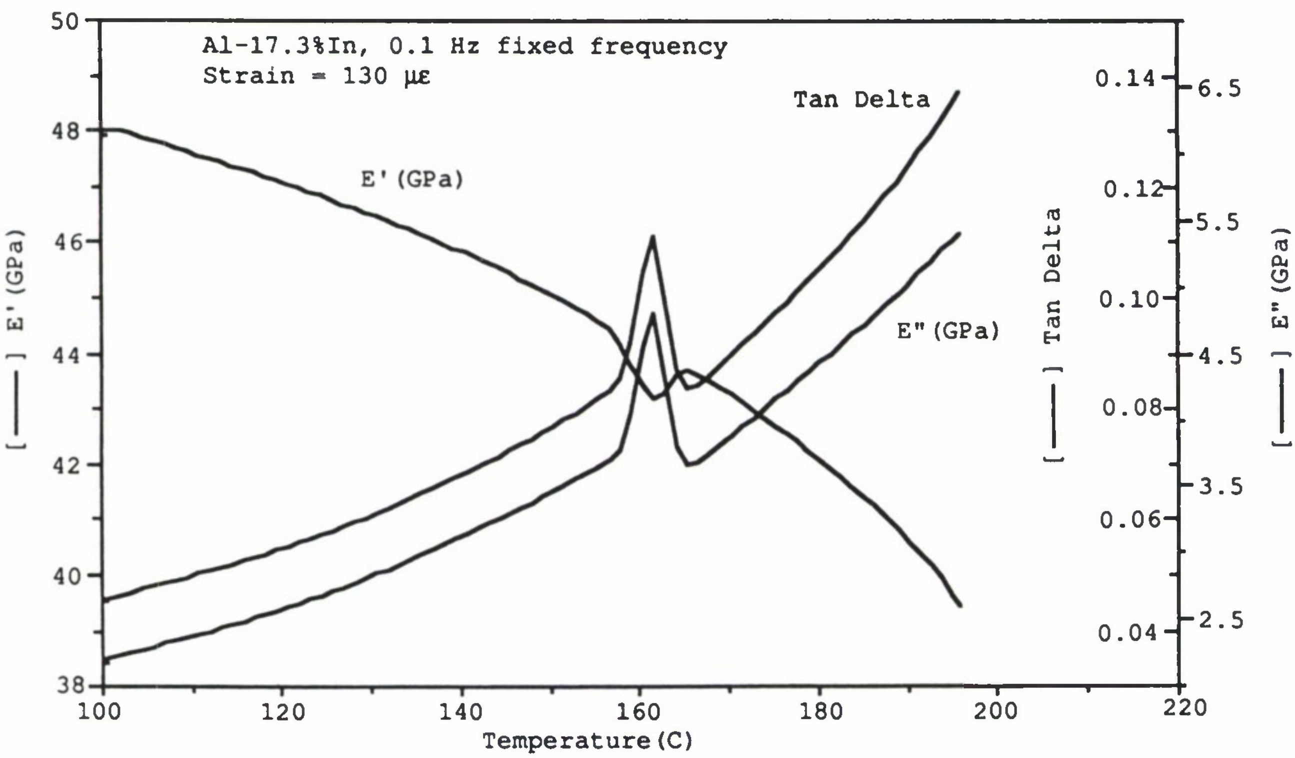


Fig. 11. DMA results for the Al–17.3 In alloy at a frequency of 0.1 Hz demonstrating a larger damping peak to background ratio at lower frequencies.

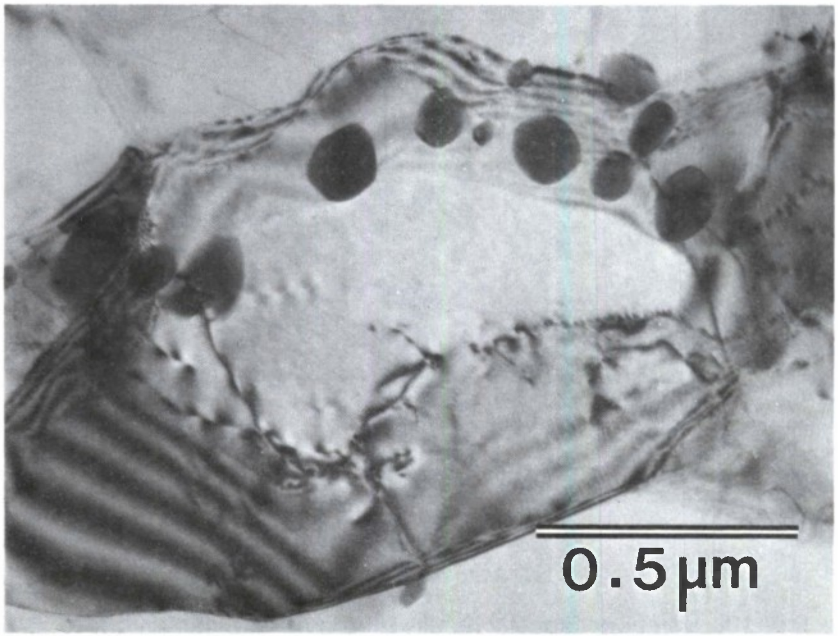


Fig. 12. Bright field transmission electron micrograph of the Al-0.6 In alloy sowing indium particles on a subgrain boundary.

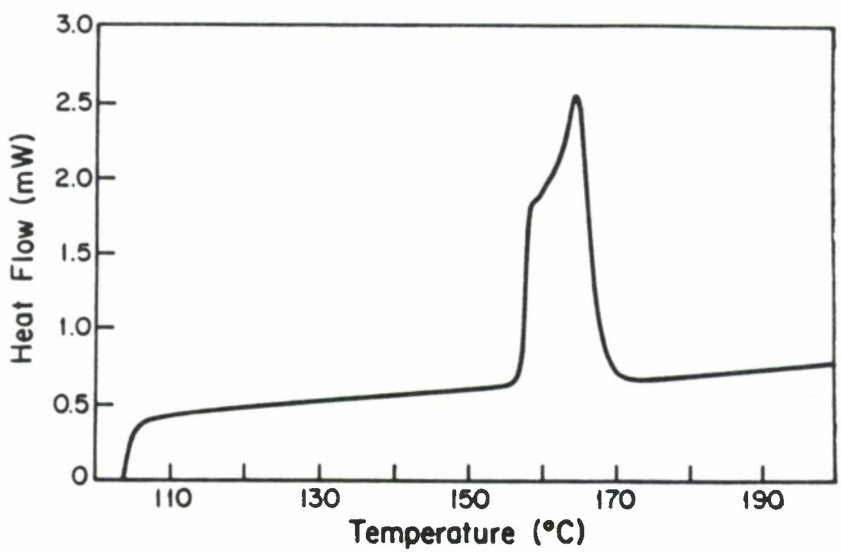


Fig. 13. DSC results for the Al-12 In alloy indicating a second melting peak due to a fine structure of indium particles which melt at a higher temperature.

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| 5 | 281 | (Gudas) |
| 5 | 2812 | |
| 10 | 2812 | (CRW) |
| 1 | 283 | (Singerman) |
| 1 | 284 | (Fischer) |
| 1 | 2844 | |
| 1 | 522.1 | |
| 1 | 522.2 | |
| 2 | 5231 | |