

The study of internal friction in metal–metal layered materials

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Abstract

Internal-friction and modulus measurements were made on 99.999% pure Al samples of stabilized grain structure and on the samples covered by a Cu or Zn layer produced by electrodeposition. These layered samples were heat treated in order to cause diffusion of the foreign atoms into the Al mainly along the grain boundaries. The usual grain boundary relaxation was decreased or suppressed by the foreign atoms but the Cu turned out to be more effective in this sense. For sufficiently thick Zn layers, even a new relaxational peak appeared. The essential role of the grain boundary dislocations can be concluded from the unusual isothermal results observed in the grain boundary relaxation temperature range, and from the amplitude dependence of the layered samples after heat treatment. The role of the covering Zn or Cu layer in the dynamic recrystallization under axial stress was also investigated.

1. Introduction

The first measurements on the grain boundary (GB) internal-friction (IF) relaxation peak were made by Ke [1] in 1947. Since then there have been many papers published to elaborate the basic mechanisms of this relaxation, the effect of solute atoms, the amplitude dependence of main peak characteristics, the role of lattice, and GB dislocations. It has been suggested that the solute atoms hindered the sliding along the smooth regions between two neighbouring ledges by segregation. The concept of “solvent” and “solute” peaks has also appeared; the GBs have been classified as solute sensitive and solute insensitive.

The effect of some impurities (Sn, Cu and Ag) having various solubilities in Al was investigated by Iwasaki [2–4]. The segregation of solute atoms along GBs and dislocations was found to be the main mechanism affecting the relaxation character. The extent of segregation depended on the solubility limit; the lower the solubility, the more atoms are segregated at GBs. It has been shown that the peak temperature and the activation energy are higher, and the peak height lower than those of pure Al.

The aim of our paper is to show the changes in the conventional GB peak of high purity Al caused by Zn and Cu interdiffusion. These investigations are interesting because (a) they can provide a good basis for understanding the damping of more complex metallic composite structures and (b) they can clarify the basic processes of the GB damping caused by solute atoms.

2. Materials and experiments

The nominal 99.999% Al wire of diameter 0.8 mm and with a stabilized structure had a mean grain diameter of about 0.01 mm. The Al wire after pre-heat treatments was put into an inverted torsion pendulum and was then annealed *in situ* at 600 °C for 1.5 h. Then, measurements were made during the heating and cooling periods between room temperature and 400 °C so that we could stabilize the grain structure. The structural stability from the viewpoint of the IF measuring technique was defined so that the spectrum remained unchanged during the next heating or cooling period. The number of periods required to ensure that the spectrum remain unchanged was always 3.

The usual frequency and amplitude of the measurements were about 0.3 cycles s^{-1} , and $\epsilon=10^{-5}$ respectively, but some measurements were made in the frequency range 0.05–5 cycles s^{-1} and in the amplitude range 10^{-5} – 25×10^{-5} . Most measurements were done under no axial stress but, in some cases, static loading up to 5 MPa was applied. The usual heating and cooling rates (when measuring spectra) were about 3 K min^{-1} .

The covering Cu or Zn layer was produced by electrodeposition on the surface of the stabilized and measured Al sample. In order to have a sufficiently good layer, a short-time electroless deposition was applied first. The layer thickness was 6 or 40 μm .

The time and temperature of the heat treatments (HTs) were calculated to fulfil the conditions of the so-called regime C or B concerning the ratio of the GB to volume diffusion [5]. As can be seen in Fig. 1,

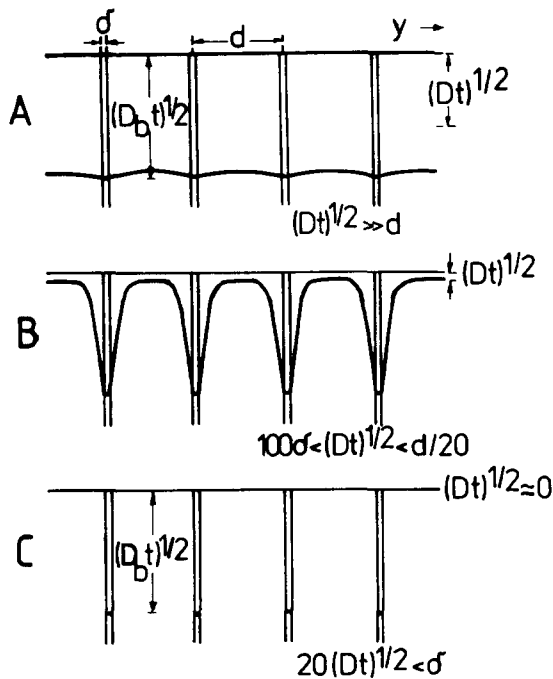


Fig. 1. Concentration profiles after diffusional heat treatments where the volume diffusion (regime A) or the GB diffusion (regime C) is dominant, or in the mixed case (regime B).

HT at sufficiently low temperatures or sufficiently short times causes the majority of the GB diffusion because of the difference between the GB diffusivity D_b and the volume diffusivity D [6]. At higher temperatures, regime A exists where the volume diffusion is fast enough and the diffusing covering material can spread out from the GBs into the volume and a nearly uniform concentration will be obtained. We preferred the C-type profile because investigation of the effect on the GBs was the aim of the work.

3. Results and discussion

3.1. Effects of diffusion on the spectra

Without any HT, the Cu or Zn layer usually caused a significant increase in the low and high temperature IF background. The GB relaxation peak always appeared but at a slightly higher temperature and the relaxation strength was reduced (Fig. 2). The characteristic data of the GB relaxation at 0.3 cycles s^{-1} are summarized in Table 1. The obvious influence seems to be rather surprising but it should be mentioned that the absence of any HT is not strictly valid because of the HT during the first measurement itself. For fairly thick Zn layers (40 μm), the appearance of a new peak at about 100 $^{\circ}C$ can be observed. This is probably caused by the GB relaxation inside the Zn layer [7].

The HT before the first measurement of the covered sample always had a significant effect on the spectra.

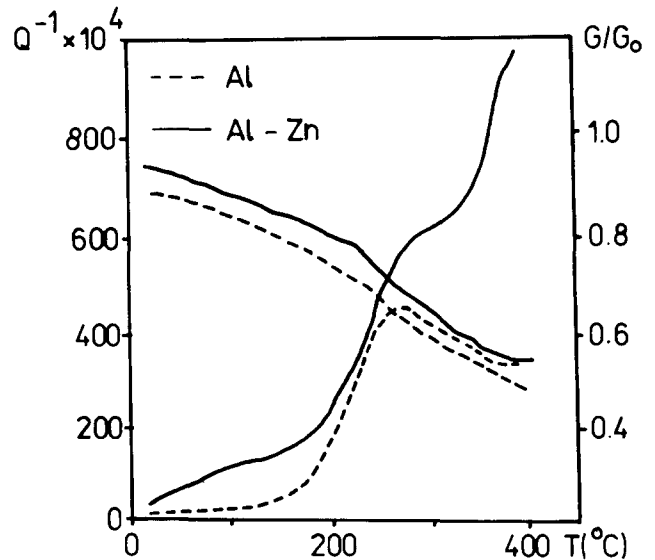


Fig. 2. The effect of a Zn layer without any HT.

TABLE 1. Changes in the temperature and height of the grain boundary relaxation peaks due to the Zn layer

	T_p ($^{\circ}C$)	$Q_{GB}^{-1} \times 10^4$
Al	270	300
Al-Zn	280	140

In the case of Al-Zn the HT at 350 $^{\circ}C$ for 2 h caused an increase in the peak temperature T_p and a considerable decrease in the peak height. This last effect is much more pronounced after HT for 4 h at the same temperature. The above effect can be attributed to the appearance and segregation of Zn atoms at the GBs which hinders GB relaxation. This statement is supported by the measurements at different frequencies. From the shift in T_p , the activation energy of the GB relaxation is evaluated to be 1.43 eV, which is considerably larger than the usual value for pure Al [8]. In the case of a Cu layer on Al wire, the HT (450 $^{\circ}C$ for 2 h) caused the disappearance of the original GB peak (Fig. 3). This HT is equivalent to the HT of Al-Zn in the sense of GB diffusion depth due to the slower diffusion of Cu. This complete elimination of the original GB peak could be connected to the low solubility of the Cu (compared with Zn) and to the appearance of precipitates on the GBs.

As has been mentioned, in the case of non-heat-treated covered samples a small relaxational peak appeared at about 100 $^{\circ}C$ owing to the fairly thick Zn layer. After HT, this peak was also detectable.

In order to determine whether the above GB peak modification effects are really due to the diffusing foreign atoms (along the GBs) or are possibly due to the

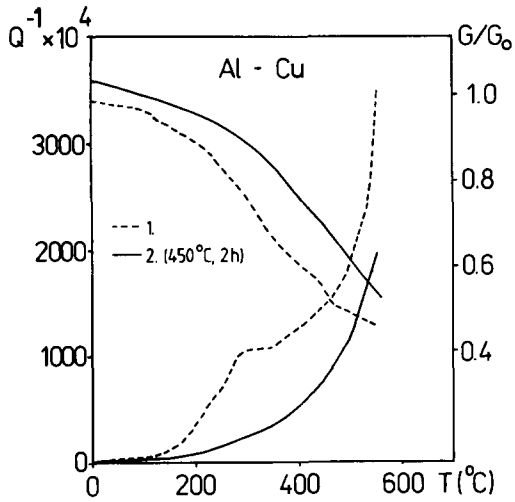


Fig. 3. The spectra obtained on Cu-covered Al wire without (curve 1, ---) and after HT (curve 2, —).

covering layer, measurements were made on samples from which the layer had been removed (by dissolution in HNO_3). The layer was dissolved after the usual HT, thereby inducing partial diffusion in the Al wire. For Al-Cu samples we observed the same result as before the dissolution, but for Zn-covered samples the GB peak seemed to increase. These different behaviours could be attributed to the different solubilities and diffusion effects of Zn and Cu in the Al matrix. The Cu, which forms GB precipitates, can have a stable effect but, in the case of the Zn which has a higher solubility and faster diffusion, the concentration of the Zn atoms on the GB can decrease gradually.

In order to investigate the dynamics of the diffusion effects, isothermal measurements were made on the Cu or Zn-covered samples just after the electrolytic deposition. The IF of the original Al sample does not depend on the time at room temperature, as expected, but the $Q^{-1}(t)$ curve obtained just a few minutes (5–10 min) after the electrodeposition shows a fairly large IF decrease. This effect can be partly attributed to the rearrangements of the structural defects produced in the layer during the electrodeposition or partly to diffusion of hydrogen (originating from the acidic deposition) to or along the (GB) dislocations.

In the temperature range of GB relaxation (between about 170 and 300 °C), an unusual isothermal increase was found in the first 10 min (Fig. 4) for pure Al or Al-Zn. The dynamic modulus decreased simultaneously. One possible explanation for this could be that the GBs (GB ledges) can act as intensive dislocation sources during the continuous vibration at this temperature. It should be mentioned that this type of IF increase was not observed for the Al-Cu samples in accordance with the more intensively blocking effect of the GB precip-

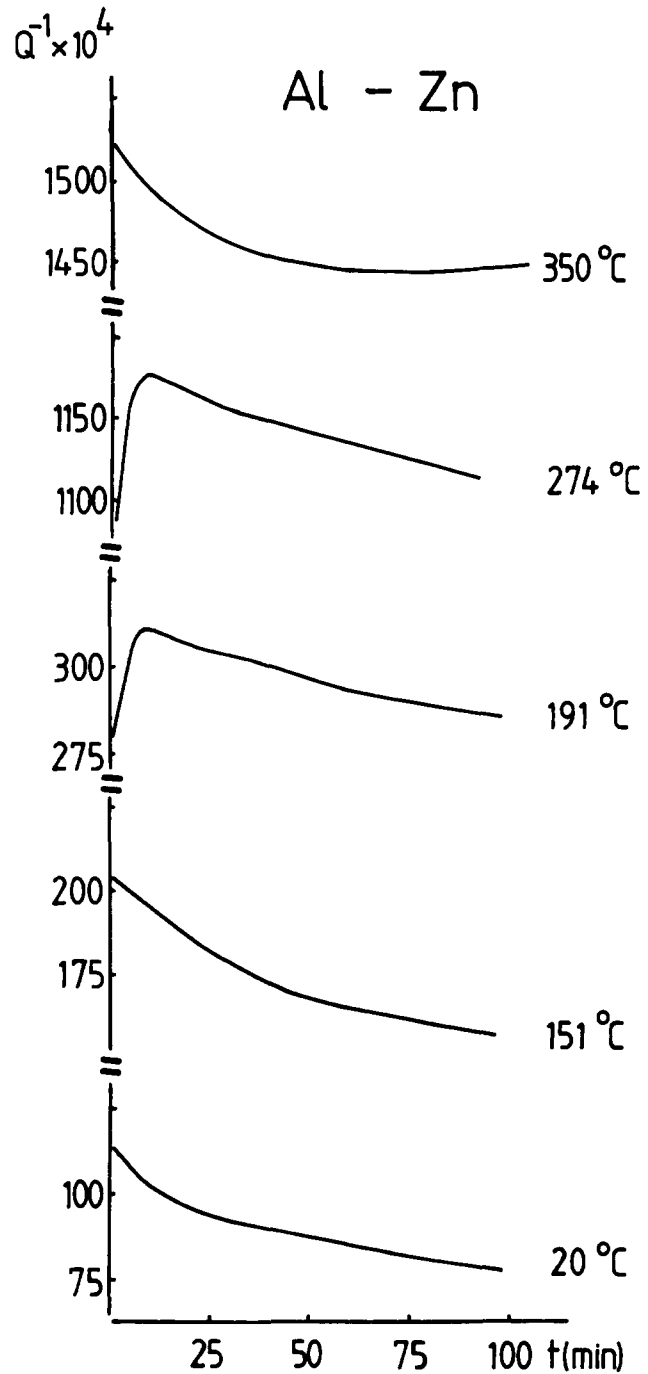


Fig. 4. Isothermal measurements at different temperatures.

itates formed in this case. These can act more effectively as blocking obstacles for dislocation sources.

3.2. Stress and strain effects

Using the possibilities of the inverted pendulum, measurements were made on the above materials at different strain amplitudes and at different loading axial stresses.

The amplitude-dependent measurements showed the usual nearly constant or slightly increasing IF (and

similarly decreasing modulus) for the increasing vibration amplitude which is the usual behaviour due to the dislocations. On measurement at a constant amplitude in this deformation range an increase in the IF was usually observed, which could be attributed to the increasing number of dislocations. At sufficiently high deformations a sudden increase in the IF was usually found, which could be attributed to the break-away of the dislocations from the pinning points. The above-mentioned behaviour (which is usual) had a surprising hysteretic character (Fig. 5) in the case of layered samples after HT. This suggests that the above type of phenomena usually attributed to the volume dislocations should be connected to the GB dislocations in this case.

In order to investigate the role of blocked dislocations and the role of the covering Zn or Cu layer, measurements were made at different axial stresses. The basic effect can be seen in Fig. 6 where the IF and the modulus of the stabilized pure Al are shown in

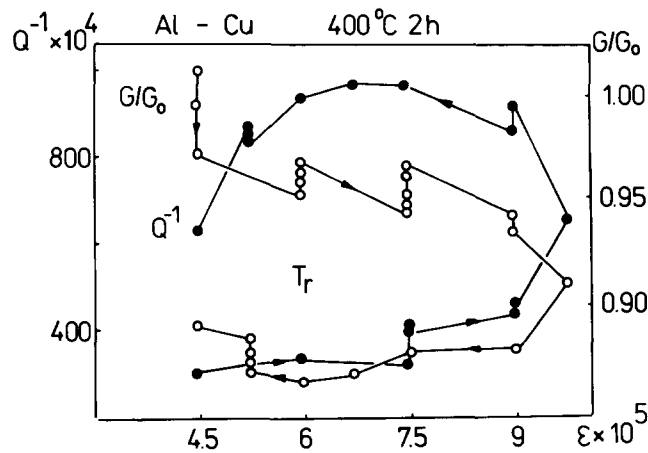


Fig. 5. Amplitude dependence measured at room temperature.

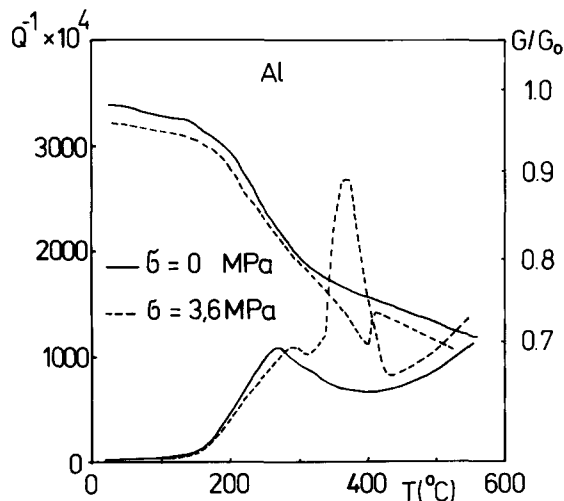


Fig. 6. Pseudopeak due to dynamic recrystallization under axial loading.

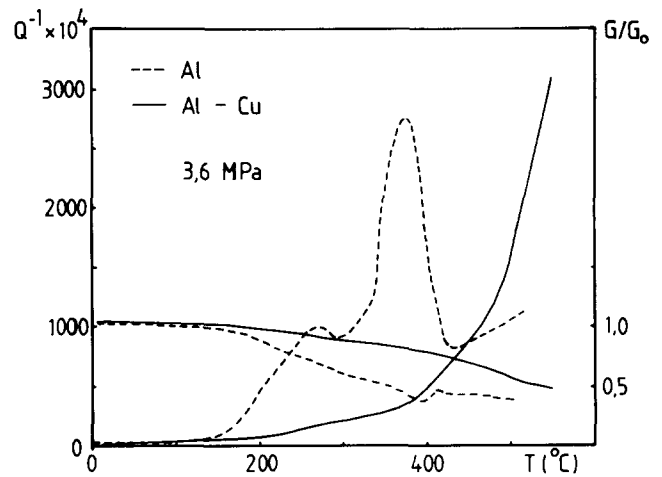


Fig. 7. No dynamic recrystallization on the copper-covered sample under axial stress.

the usual circumstances ($\sigma=0$ MPa) and under axial load. In the loaded conditions, a very large non-relaxational irreversible pseudopeak can be seen between 370 and 400 °C, accompanied by a modulus increase. The above irreversible dynamic recrystallization of the earlier stabilized structure does not appear in the covered samples in the investigated stress range, as can be seen for the Cu layer in Fig. 7. The reason for the absence of the earlier effect could be the presence of the Cu covering layer which can block the recrystallization owing to its fairly large modulus, hindering the size change during the transition. Another reason for the above-mentioned behaviour could be the blocking effect of the Cu atoms on the GBs or GB dislocations. In order to investigate whether the first external or the second internal effect caused the absence of the dynamic recrystallization in the covered samples, measurements were made after removal of the Cu layer. A fairly large pseudopeak accompanied by a modulus increase was observed again. The peak monitoring the irreversible structural change is similar to that observed in pure Al but its temperature is about 100 °C higher, and no GB relaxation peak appeared at lower temperatures. The above phenomenon shows that the influence of the Cu layer must be essential in the disappearance of the dynamic recrystallization in the covered material. On the contrary, the temperature shift of the pseudopeak which reappears shows that some of the Cu atoms which diffused along the GBs remain in this site, hindering the dynamic recrystallization.

4. Summary

The influence of foreign atoms diffusing along the GBs from the outer layer of the sample has a fairly

large effect on the GB relaxation of the Al matrix. This effect can be rather different depending on the solubility and on the diffusivity of the material in the layer. This phenomenon is not caused by the physical effect of the covering layer but the diffusing atoms affect the GB relaxation as can be concluded from the results after removal of the layer. On the contrary the covering layer can have a significant effect on the dynamic recrystallization, which is shown by the reappearance of the transformational pseudopeak after removal of the layer. The most valuable result is found from the investigations made at different amplitudes where the hysteretic $Q^{-1}(\epsilon)$ dependence appeared in the case only where the covered sample was heat treated. However, owing to this HT the foreign atoms diffuse mainly along the GBs. This means that a significant

part of the amplitude-dependent background could be caused by the GBs or by the GB dislocations.

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