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1990 J. Phys.: Condens. Matter 2 9247

(http://iopscience.iop.org/0953-8984/2/47/002)

J. Phys.: Condens. Matter 2 (1990) 9247–9256. Printed in the UK

Lubrication agents of dislocation motion at very low temperature in cold-worked aluminium

J Lauzier†, J Hillairet†, G Gremaud‡ and W Benoit‡

† Département de Recherche Fondamentale, Service de Physique, Centre d'Etudes Nucléaires, 85X, 38041 Grenoble Cédex, France

‡ Institut de Génie Atomique, Ecole Polytechnique Fédérale, Ecublens CH 1015 Lausanne, Switzerland

Received 3 May 1990, in final form 26 July 1990

Abstract. Measurements of the dynamic modulus and internal friction of ultrahigh purity aluminium were performed after low-temperature plastic deformation. The salient result is that the low-temperature deformation provokes an important decrease of the elastic modulus and a simultaneous disappearance of the Bordoni relaxation. These results confirm the existence of a lubrication process proposed recently to explain the very small value of the critical resolved shear stress observed at low temperature in FCC metals.

Further insight into the nature of the defect species responsible for this lubrication has been attained by comparing the results obtained after low-temperature irradiation and those following low-temperature plastic deformation and subsequent anneal sequences at different temperatures. Vacancies are believed to be the defect species responsible for the lubrication.

1. Introduction

The experiments on the elastic limit in pure face-centred cubic (FCC) metals suggest that the mobility of dislocations in these metals remains high, even at very low temperatures. This result is in apparent contradiction with internal friction (IF) measurements [1], and more precisely with the usual interpretation of the Bordoni relaxation (BR) by the kink pair formation (KPF) mechanism. Indeed, this interpretation implies that the mobility of the dislocations should be strongly decreased when temperature goes down because the KPF mechanism no longer applies.This discrepancy between the interpretation of the BR and mechanical testing has led to some doubt about the interpretation of the BR by the KPF mechanism.

Ultrasonic experiments have been performed with the low-frequency–ultrasounds (US–LF) acoustic coupling method [2] in order to specify with a higher reliability the mechanism responsible for the IF relaxation peaks. The principle of the US–LF coupling method consists in measuring variations of attenuation α and velocity v of ultrasonic waves in a sample subjected to a permanent sinusoidal low-frequency applied stress σ. The closed curves $\Delta\alpha(\sigma)$ and $\Delta v/v(\sigma)$, measured during one cycle of the low-frequency stress, are drawn at different temperatures. The shape of these curves and their evolution as a function of temperature exhibit specific features for every mechanism controlling dislocation motion [2]. In the case of the BR, the experimental evidence is that the real curves look very similar to the calculated one for the KPF mechanism [3]. These results strongly support the interpretation of the BR in terms of the thermally activated KPF mechanism. Moreover, using the same experimental technique, it has been observed that a small additional plastic deformation at low temperature (0.1% at 10 K) changes drastically the shape of these curves [3]. To interpret these results it was assumed that point defects are created during the plastic deformation and that these defects activate a lubrication process for the dislocation motion [3, 4]. This conclusion is very important because it bridges the gap between the interpretation of the BR by the KPF mechanism and the very small value of the critical resolved shear stress observed at low temperature.

More recently a second series of experiments was initiated both to confirm and to obtain a better understanding of this lubrication process, based on the realization of low-frequency IF and modulus measurements with the use of a vibrating reed system. Two very distinct conditions can be used. First, irradiation with fast electrons at very low temperature, to selectively create elementary point defects, and subsequent anneals have been presented in a recent paper [5]. The salient result is that the introduction of point defects on the existing dislocations during irradiation at very low temperature provokes a substantial decrease of the elastic modulus in agreement with the lubrication process as proposed in [3, 4]. In this second paper results obtained with the same experimental approach but after plastic deformation and subsequent anneals are presented. The expected behaviour of the modulus defect inferred from a lubrication process is presented on figure 1. Curve 1 delineates the modulus profile observed after room temperature deformation. The change in the modulus (a) appearing between 50 and 200 K is due to the BR process. After a small low-temperature deformation, the lubrication mechanism should induce an important decrease of the low temperature elastic modulus (b). This important softening should be associated with the disappearance of the modulus change due to the BR (c). A recovery effect is also expected when the point defects under consideration become annealed (d).

2. Experimental method

Measurements of dynamic modulus and internal friction of ultrahigh purity aluminium were performed using a vibrating reed system, in which samples were rectangular plates 15 mm long, 5 mm wide, with a thickness of 200 μm, obtained by spark erosion. These strips were clamped in a copper block in which a heat exchanger was incorporated. This system can be used down to 4.7 K, the temperature of liquid helium at a pressure of 1.2×10^5 Pa. A Rh–Fe resistance thermometer was used to monitor the sample temperature. For the measurement of damping and dynamic modulus, the reeds were electrostatically vibrated in vacuum, to produce flexural vibrations with a typical amplitude of 10^{-6}, in the frequency range of 800 Hz. An automatic data acquisition system enabled direct recording of the relevant parameters: frequency, f, damping, Q^{-1}, and absolute temperature, T. It should be recalled that the relative elastic modulus values are proportional to the square of the relative frequency values [6].

After mounting, *in situ* cold-working was produced by mechanical bending at different deformation levels. It was preceded or followed by various *in situ* thermal treatments.

The studied material was ultrahigh purity aluminium prepared by zone melting at the Centre d'Etudes de Chimie Métallurgique de Vitry sur Seine (France). Spectrometric analysis and neutron activation both indicate that the total impurity concentration of this aluminium is well below 1 at. PPM [7].

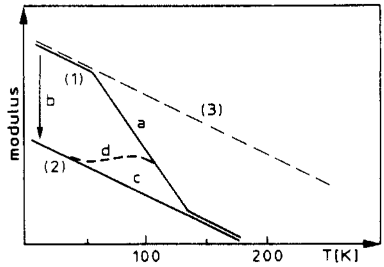

Figure 1. Expected behaviour of the modulus inferred from a lubrication process of the dislocations, generated by cold-working: curve (1) modulus behaviour observed after room temperature deformation, curve (2) modulus behaviour expected after low-temperature deformation, curve (3) theoretical lattice modulus behaviour.

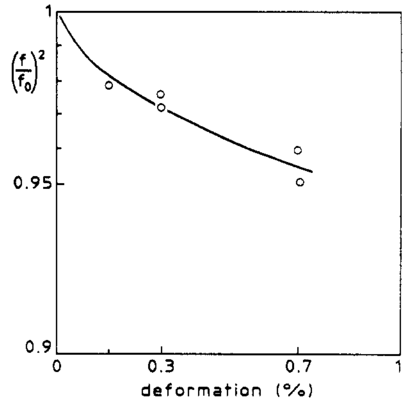

Figure 2. Softening induced by cold-working of the sample and observed by the modulus decrease at 4.7 K. This softening depends strongly on the amount of plastic deformation.

Experiments were also carried out on samples of the same ultrahigh purity aluminium, but after doping with hydrogen, in order to determine the effect of extrinsic interstitial point defects. Doping was obtained by implantation of protons with varying energy to get a uniform concentration over the whole sample thickness.

3. Observation of a softening induced by cold-working

The modulus variations found after cold-working at 4.7 K at different deformation levels are reported in figure 2. These experimental results show that an important softening of the samples takes place as a result of the plastic deformation at low temperature and that this softening depends strongly on the deformation level. It is interesting to note that the modulus decrease induced by cold-working at 4.7 K is of the same order of magnitude as the modulus defect associated with the Bordoni relaxation, when this relaxation is measured after cold-working at 4.7 K, followed by annealing at 290 K (figure 3). This leads to the idea that the same dislocation network is responsible for these two modulus effects, as has been already proposed to explain the results obtained by the US-LF coupling technique [4]. This is also in very good agreement with the experimental results obtained by the US-LF coupling method, by which it is observed that the mechanism responsible for the cold-work-induced softening is completely replaced by the mechanism of thermally activated KPF (responsible for the Bordoni relaxation) after an annealing of the sample at 200 K [3].

After cold-working, measurements were performed during a linear increase of the temperature, at a rate of 2 K min^{-1}. Curve a on figure 4 represents the reference state of the sample, obtained after an *in situ* annealing at ≅360 K. Curves b, c, d, e and f present the modulus variations obtained after the samples have been annealed at 360 K

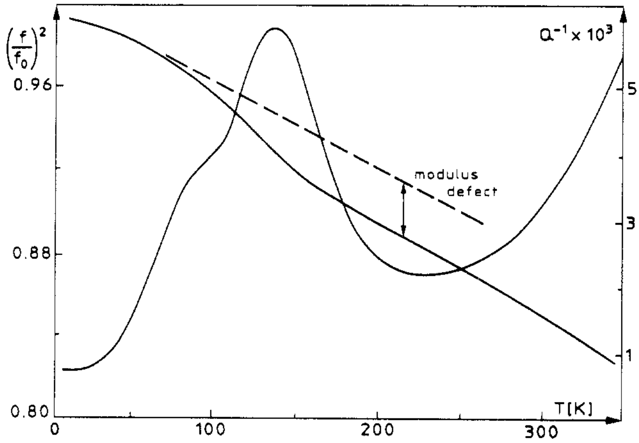

Figure 3. Measurement of the Bordoni relaxation in a sample, cold-worked at 4.7 K (0.3%) and annealed at 290 K, showing the modulus defect of the BR which can be measured, by drawing the theoretical lattice modulus without BR (broken line parallel to the experimentally measured modulus). The estimated modulus defect is of the same order as that which is measured for the softening induced by cold-working (see figure 2).

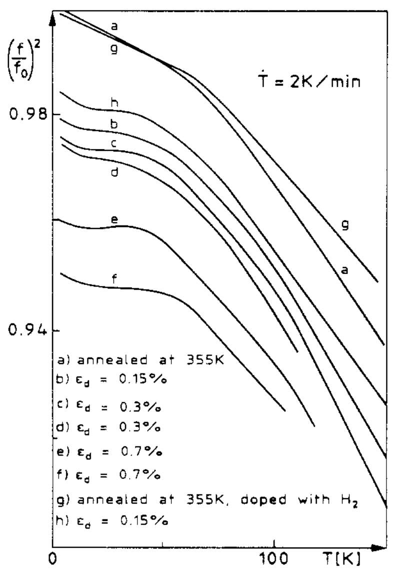

Figure 4. Modulus variations after an annealing at 355 K (a), followed by different levels of plastic deformation at 4.7 K (b, c, d, e and f), and showing the effect of softening induced by the cold-working. Similar results are obtained in hydrogen-doped samples (g, h).

and cold-worked at 4.7 K at different estimated maximum strain at their embedding (0.15%, 0.3% and 0.7%).

All the variations in modulus as a function of measurement temperature exhibit similar patterns (figure 4, curves a, b, c, d, e and f). It is also noticeable that the softening induced by cold-working at 4.7 K is not enhanced when the temperature is increased. On the contrary, the modulus seems to evolve with temperature towards a return to its initial value. This will be analysed in detail in the next section.

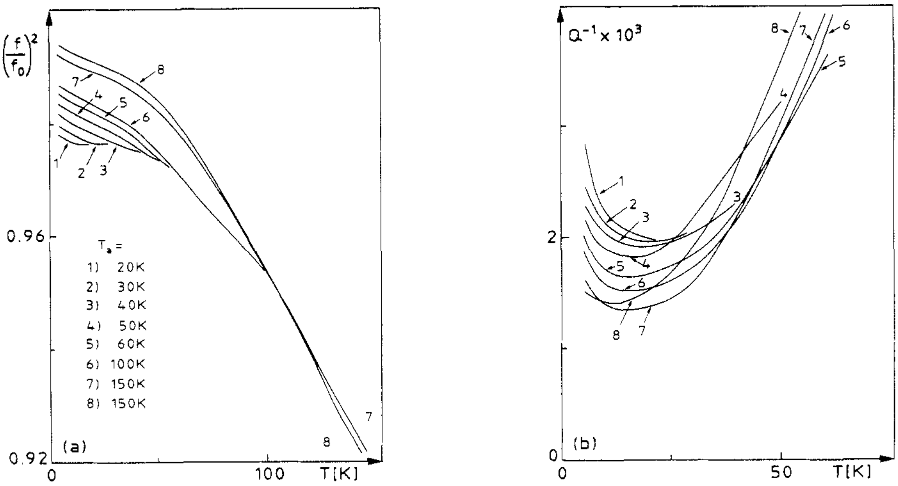

Figure 5. Measurements of the annealing of the cold-worked (0.3%) induced softening (*a*). The internal friction presents a quick increase in the very low-temperature range (*b*), which is also progressively annealed by increasing the temperature.

In figure 4, curves g and h present the same effect of softening when the measurements are performed in hydrogen-doped samples. Curve g is the reference curve obtained without cold-working, and curve h shows the change in modulus after an estimated deformation of 0.15% performed at 4.7 K. It is clearly seen that the hydrogen doping of the sample does not inhibit the softening effect. It remains comparable to that in undoped specimens.

4. Annealing of the softening induced by cold-work

The above-mentioned softening, as observed at the cold-work temperature, can be annealed by increasing the temperature of the sample. This effect is shown in figure 5(*a*) by successive sawtooth-like annealings performed between 20 and 150 K. During these experiments, the internal friction exhibits a rapid rise towards the very low temperatures. This increase could be the high-temperature side of a relaxation peak whose maximum occurs at a temperature lower than 4 K (figure 5(*b*)). These experiments show that the softening induced by cold-work is progressively annealed as the temperature rises (figure 6). At the same time, the increase of internal friction in the very low-temperature range is also progressively annealed, in a very similar manner. It is important to note that neither the recovery of the modulus, nor that of the low-temperature internal friction shows the sudden well defined recovery stage which is observed at 50 K in the case of electron irradiated samples (see figure 9 below). On the contrary, it proceeds very smoothly between 4.7 K and 150 K, as can be noted in figure 6.

Beyond 200 K, the softening induced by the cold-work appears to be entirely annealed. This is shown in figure 7. Curves 1 represent the effect of temperature on the modulus and the internal friction just after an *in situ* annealing at 360 K. In this reference state, the sample presents a relatively small Bordoni relaxation peak and associated modulus defect. Just after the cold-working performed at 4.7 K, a first increase of

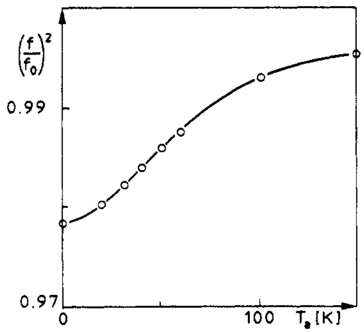

Figure 6. Plot of the progressive disappearance of the cold-worked (0.3%) induced softening, measured at 4.7 K, as a function of the maximum annealing temperature T_a attained in figure 5.

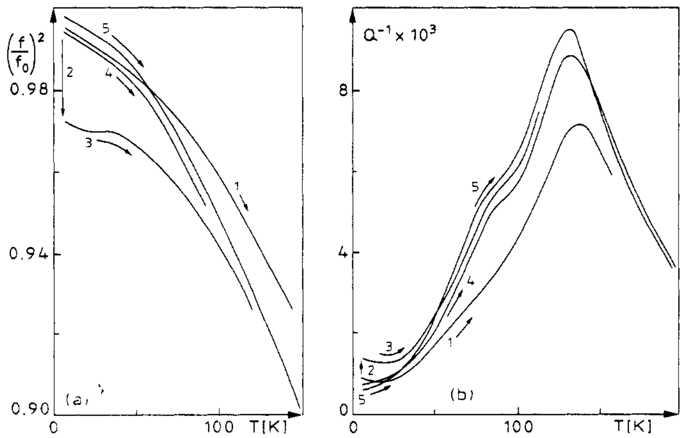

Figure 7. Measurements of the modulus (a) and the damping (b) in a sample annealed at 355 K (curves 1), cold-worked at 4.7 K (0.3%), and then measured several times by increasing the temperature to several different maximum annealing temperatures (curves 3, 4 and 5). One can see the softening effect on curve 3, and the annealing of this softening on curves 4 and 5. The Bordoni relaxation has been emphasized by the cold-working (curve 3), but also by the successive annealings (curves 4 and 5).

temperature between 4 and 120 K, represented by curve 3 in figure 7, exhibits the effect of softening at 4.7 K, but also the effect of annealing of this softening at higher temperatures, very similar to the more detailed measurements presented in figure 5(a). During this increase of temperature, the internal friction, illustrated by curve 3 in figure 7(b), shows the low-temperature side of a well developed Bordoni relaxation peak. During a second temperature rise from 4.7 to 200 K consideration of the modulus curve (curve 4 in figure 7(a)) reveals that the modulus defect associated with the Bordoni relaxation is enhanced, compared to that in the undeformed sample (curve 1). The internal friction also presents a more marked Bordoni relaxation peak. During the last increase of temperature, (curves 5 in figures 7(a) and 7(b)) the modulus at 4.7 K is once again increased, and this effect is associated with the more developed Bordoni relaxation peak.

Similar measurements were also performed in a hydrogenated sample. The results obtained, reported in figures 8(a) and 8(b), clearly show that the hydrogen doping,

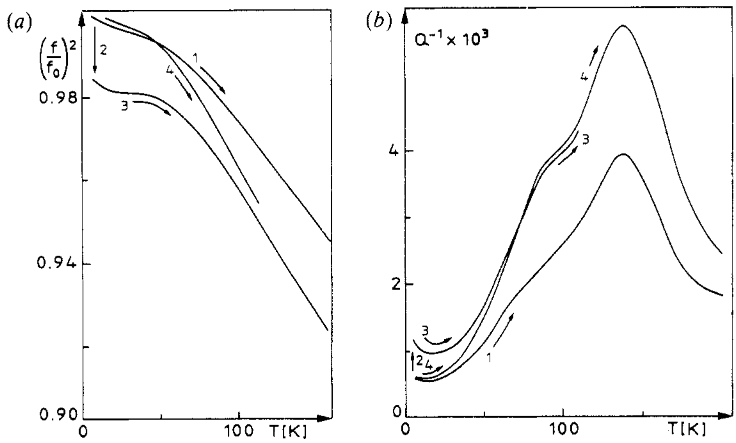

Figure 8. Measurements of the modulus (*a*) and the damping (*b*) of a sample doped with hydrogen (curve 1), cold-worked at 4.2 K, and then measured several times (curves 3 and 4). Very similar results to those measured in undoped samples (figure 7) are obtained.

responsible for the presence of extrinsic interstitial point defects in the metal, does not influence the mechanisms associated with the softening induced by cold-working.

5. Analysis of results

The good agreement between the experimental results and the calculated results predicted on figure 1 is remarkable. The important increase of the dislocation mobility observed by the LF coupling method is effectively correlated with an important softening observed by internal friction after cold-working at 4 K. The results of the US-LF coupling method clearly shows that this softening is connected with a decrease of the BR mechanism. In the present experiments the decrease of the amplitude of the BR can occur only partially, mainly because annealing already takes place at temperatures lower than those in the range of the BR. Taking into account these annealing effects, there is little doubt that the observed low-temperature softening is correlated with a substantial decrease of the BR.

As already stated, an important increase of the IF appears at temperatures below 10 K (figure 5(*b*)). This effect could also be related to results obtained by the US-LF coupling technique, after low-temperature deformation ('smile signatures' in [1]). These results have been explained by the presence of mobile point defects in the vicinity of the dislocation core [1], which could be the same point defects as those responsible for the lubrication process. But this increase of the damping could also be due to the high-temperature side of a relaxation peak due to a thermally activated mechanism of kink migration along the dislocation lines.

6. Discussion

As shown in the last section the effects observed in the cold-work experiments are very similar to those expected (figure 1). Yet the proposed interpretation of a lubrication mechanism is not evident. Effectively, at first sight the more trivial interpretation should be that the low-temperature plastic deformation induces a depinning of dislocations from pinning points, and that this depinning is responsible for the decrease of the modulus and the increase of the damping. However, this classical interpretation seems to be incorrect for at least three reasons:

(i) If depinning occurs, the decrease of the 4 K modulus should be associated with an increase of the BR. On the contrary, the BR modulus defect decreases as the 4 K modulus is decreased, which certainly means that the low temperature deformation prohibits the BR mechanism.

(ii) The decrease of the 4 K modulus is also observed during low-temperature irradiation [5].

(iii) Depinning effects could not explain the shape of the curves observed at low temperatures with the US–LF coupling technique [1], after low-temperature deformation.

As a consequence, the strong decrease of the modulus observed at 4 K after a small plastic deformation is most probably linked to some type of cold-work produced defects which lubricate the dislocations. More precisely, these defects would short-circuit the KPF mechanism as proposed in [4].

It has been also suggested that the experimental procedure for producing plastic strain, which involves plastic bending, is known to generate significant internal stress levels in the material. And it is known that a polarising stress reduces the height of the Bordoni peak. The possibility of this occurring for the observed decrease of the Bordoni peak height after cold-working can be eliminated for at least three reasons:

(i) It cannot explain the modulus softening observed below the Bordoni peak temperature.

(ii) It cannot explain the similar softening observed during irradiation.

(iii) The plastic bending used here is very small (0.3% of plastic deformation) and leads probably to very small polarising stress fields.

Concerning the nature of the defects, both the US–LF coupling observations [3, 4] and the measurements made after electron irradiation [5] favour the role of point defects. It has been proved that cold-work essentially creates vacancies [8]. Consequently defects of this type should be responsible for the observed softening. This interpretation is confirmed by the results on an electron irradiated sample (figure 9). During irradiation at 5.7 K a substantial softening is observed. It disappears during annealing at 50 K, which corresponds to stage I_E of the residual electrical resistivity recovery. The interpretation is straightforward: the low-temperature irradiation creates interstitials and vacancies around the dislocations. The vacancies are responsible for the lubrication process. During stage I_E, interstitials migrate to the vacancies and this results in their mutual annihilation. The softening disappears and stage I_E corresponds to a hardening stage. The results of figure 9 show another effect. On the fifth curve a relaxation effect is observed at 45 K (relaxation peak and modulus defect). As this effect disappears during stage I_E it could be due to an interaction mechanism between the dislocations and moving interstitials located around the dislocations (before they reach the dislocations and the vacancy sinks).

One more problem remains to be considered concerning the very large temperature extent of the annealing (figure 5). This feature is also characteristic of electrical resistivity measurements made during the annealing of cold-worked FCC metals. These electrical

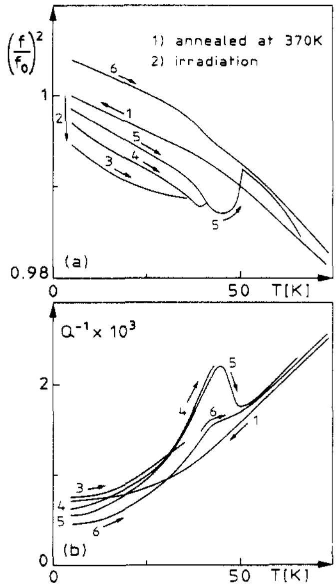

Figure 9. Measurements of the annealing of an electron irradiation-induced softening [5]. In comparison with the annealing of the cold-work-induced softening (figure 5), the annealing of the electron induced softening presents a marked effect at the temperature of stage I_E of the residual electrical resistivity recovery. This marked effect beginning by a small modulus decrease just before the quick annealing of the softening (curve 5) is correlated with a maximum of internal friction (curve 5 in figure 9(*b*)).

resistivity measurements show the absence of recovery during annealing through the temperature range of stage I (0–60 K) and recovery occurs only at higher temperatures (stage II). On the other hand, figure 5 is in good agreement with the results obtained by the US–LF coupling technique, which show the same annealing temperature range for the big increase of dislocation mobility induced by the low-temperature plastic deformation (behaviour of the 'smile signature' in [4]). We conclude that experimental techniques sensitive to defects in the bulk are not able to detect annealing at very low temperature whereas experimental techniques sensitive to defects situated close to the dislocations allow recovery effects to be observed at very low temperature. Vacancy-type defects located in the vicinity of dislocations anneal in a very broad temperature range beginning at very low temperatures. This annealing seems to be due to the small motions of the dislocations around their equilibrium positions.

7. Conclusion

After low-temperature plastic deformation an important decrease of the low-temperature elastic modulus is observed. This softening is associated with the disappearance of the modulus change due to the Bordoni relaxation. These results confirm the existence of a lubrication process for the dislocation motion [1] and explain the very small value of the critical resolved shear stress observed at low temperature in FCC metals. This lubrication process is linked to the presence of vacancies on the dislocations. After plastic deformation the annealing of this softening occurs through small motions of the dislocations around their equilibrium position. After electron irradiation this softening disappears during stage I_E and this annealing is associated with the migration of the self-interstitials to the vacancies.

Acknowledgments

The authors wish to express their appreciation to G Revel, who prepared, with unique purity, the aluminium that was studied. The operating team of the low-temperature facility are gratefully acknowledged for their help in the experiments.

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