

# Influence of dislocation networks on the relaxation peaks at intermediate temperature in pure metals and metallic alloys

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## ABSTRACT

Isothermal mechanical spectroscopy (IMS) experiments were performed in a 5N aluminium single crystal after 1% cold work. A relaxation peak was observed at low frequency and at about room temperature. The activation energy:  $H_A$  and the limit relaxation time  $\tau_0$  turned out to be 0.5 eV and  $10^{-8}$  s, respectively. The peak completely disappeared after an annealing at 673 K. Transmission electronic microscopy experiments were performed in the same material at various temperatures corresponding to the ones of the damping experiments. At room temperature, the sample exhibited essentially dislocation walls. A comparison between changes in these dislocation walls and the evolution of the IMS spectra allowed to conclude that the origin of the relaxation peak was the dislocation motion inside the dislocation walls.

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## 1. Introduction

Relaxation peaks observed between 0.3 and 0.7  $T_M$  ( $T_M$ : melting point) in single crystals of pure metals [1–12] or metallic alloys [13] are attributed to a relaxation effect associated with the dislocation microstructure. Therefore, internal friction peaks observed in polycrystals in the same temperature range can also be associated with dislocation motions. However, sometimes no peak is observed in single crystals [6,8,14] or polycrystals [15,16] even though these samples contained scattered dislocations or dislocation networks. It was also found that a small cold work was necessary to make the peak appear or increase [6,13,17]. The relaxation parameters of these peaks (limit relaxation time  $\tau_0$ , activation energy  $H_A$ , and relaxation strength) depend on the thermal treatments undergone by the samples such as annealing after cold work [15]. Some authors [8,11,18,19] tried to characterize the dislocation microstructure of the samples by internal friction. In fact, the pictures obtained by transmission electronic microscopy (TEM) generally displayed various kinds of dislocation microstructures (isolated dislocations, polygonization walls, deformation cells and so on) and it was very difficult for these authors to link a particular microstructure to the observed relaxation peaks. Moreover, the damping experiments were performed at fixed frequency during continuous heating or cooling which prevented the evolution of the peaks with the thermal treatment to be described with a good accuracy.

On the contrary, isothermal mechanical spectroscopy, by measuring the internal friction in a very large frequency range when the microstructure of the sample is stabilized is a reliable technique for describing the evolution of the damping spectra for instance during heating after cold work. The comparison between the results obtained at the same temperature before and after an annealing at a higher temperature is very easy. Moreover, because these experiments are carried out at very low frequencies, the relaxation peaks appears at lower temperature than for measurements made, for instance, at 1 Hz or more. At least, TEM observations can be made exactly at the same temperature as the internal friction experiments. So, the change in internal friction spectra can be easily related to the microstructural change observed by TEM.

## 2. Experimental procedure

### 2.1. Materials

A 5N aluminium single crystal was provided by Goodfellow Company. Specimens were cut from the bar by spark machining and the surface layer was chemically removed. To introduce fresh dislocations, the specimen was 1% cold worked by torsion. Laue X-ray diagrams of the sample were taken before mounting in the pendulum. After the experiment, Laue diagrams were taken again and macroscopic etching was made to verify that no recrystallization had occurred during the high temperature annealing.

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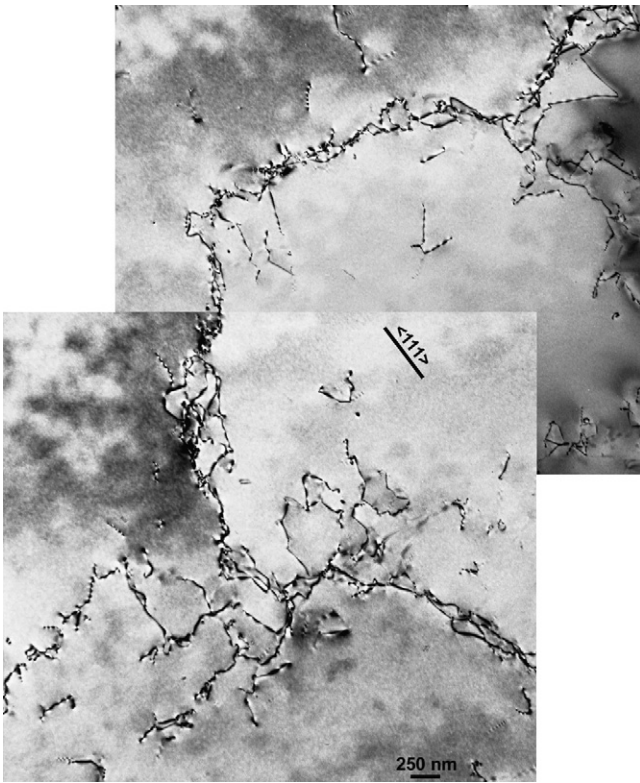


Fig. 1. Dislocation walls observed at 294 K after 1% cold work.

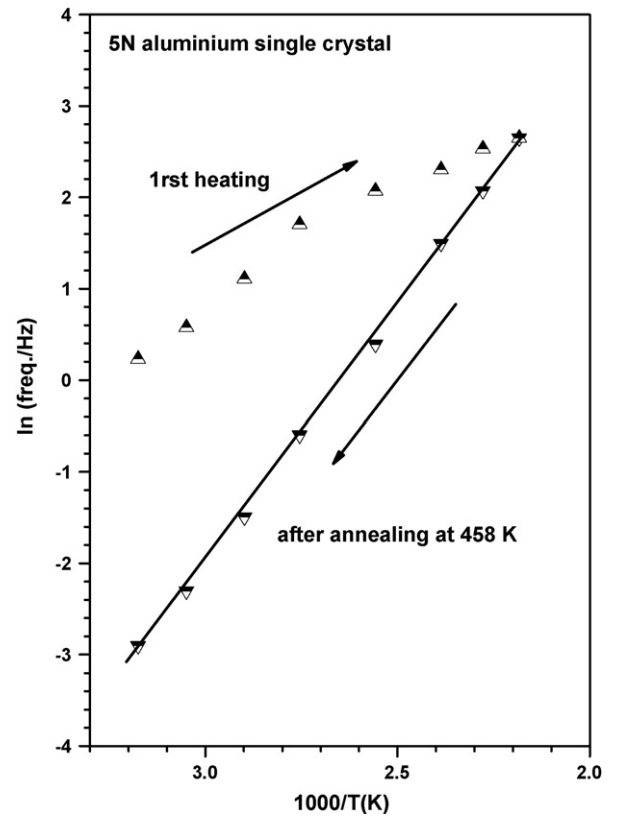


Fig. 3. Napierian logarithm of peak frequencies vs. inverse temperature for the relaxation peaks obtained during the first heating (up-triangles) and after annealing at 458 K (down-triangles).

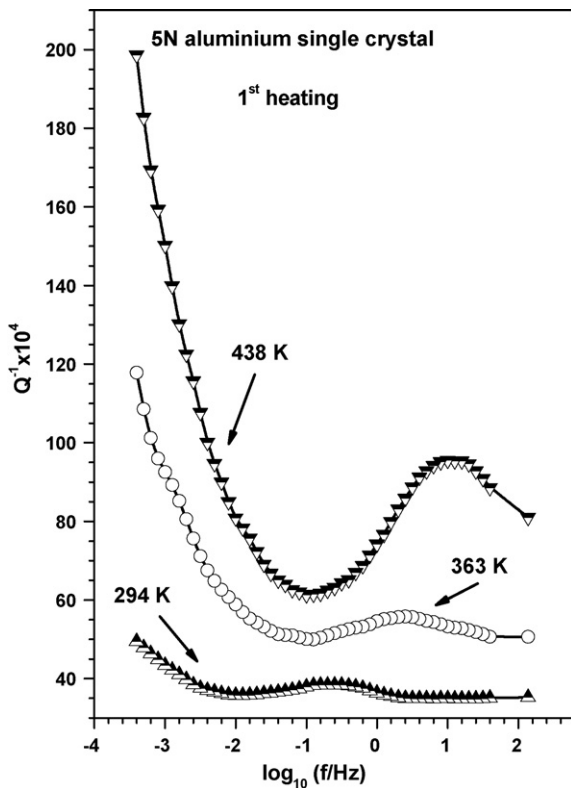


Fig. 2. Internal friction spectra obtained at 294 (up-triangles), 363 (circles) and 438 K (down-triangles) during the first heating after cold work.

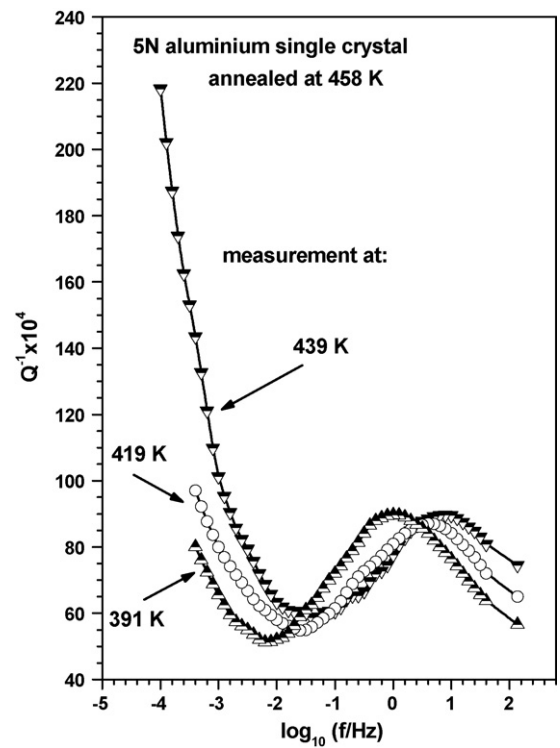


Fig. 4. Internal friction spectra obtained at 391 K (up-triangles), 419 K (circles) and 439 K (down-triangles) after annealing at 458 K.

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