The hyperbolic sine relation and dip test data in constant strain rate compression experiments

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Abstract. Single crystals of Ni₃Al, intrinsic Ge, Cu, Cu with 5at%Al and 7.5at%Al are submitted to constant strain rate compression tests and stress reduction experiments. Curves of the initial creep rate as a function of the amount of stress reduction are analysed in terms of a hyperbolic sine relation based on the thermal activation of the dislocation velocity. It is shown that this relation satisfactorily fits the curves of the two first crystals. However, this is not observed for the three last ones. Reasons for this behaviour are proposed as well as a method for the effective stress evaluation.

1. Introduction

Stress reduction experiments have been proposed [1,2] to determine the thermal component $\tau^*$ of the applied stress $\tau$. $\tau^*$ is the stress necessary for a dislocation to overcome localized obstacles. In a simple description, $\tau$ and $\tau^*$ are related by [3]:

$$\tau = \tau^* + \tau_i$$

where the internal stress $\tau_i$ represents the athermal elastic interaction with other distant dislocations.

Stress reduction experiments are usually performed in creep, see e.g. [2,4,5], much more seldom under constant strain rate tests, see e.g. [1,6], where they are more complicated to perform. The procedure can be briefly described as follows: at strain $\gamma$ on the stress-strain curve, the stress is suddenly reduced by an amount $\Delta \tau$, then the sample is allowed to creep under the stress $\tau-\Delta \tau$, for a short period of time. The initial creep rate $\dot{\gamma}_{pi}$ is recorded and observed to be positive, negative or zero depending on the magnitude of $\Delta \tau$. At zero creep rate $\Delta \tau = \tau^*$, for strain $\gamma$. The curve of $\dot{\gamma}_{pi}$ as a function of $\Delta \tau$ can be described by a hyperbolic sine function, as proposed by Milicka [5]:

$$\dot{\gamma}_{pi} = A \sinh \left[ \frac{V(\tau^*-\Delta \tau)/kT}{kT} \right]$$

In terms of the thermal activation theory, $A$ is a coefficient that depends on temperature through an activation energy and $V$ is the activation volume, $A$ and $V$ being constant during the stress dip. Relation (2) assumes i) a constant mobile dislocation density during the fast stress reduction and ii) a dislocation velocity that is thermally activated, i.e. depends on stress and temperature through the hyperbolic sine law [7]. This latter dependence is valid whatever the stress, even at very low values when dislocations exhibit forward and backward motions, provided these are likely to occur. Relation
(2) indicates that \( \dot{\gamma}_{\text{pi}} \) is positive for \( \Delta \tau \) values smaller than \( \tau^* \), is zero for \( \Delta \tau = \tau^* \) and negative for \( \Delta \tau < \tau^* \), as expected.

The point of this study is to compression deform various single crystals, record the \( \dot{\gamma}_{\text{pi}} (\Delta \tau) \) curves and to assess the Milicka law (relation (2)). The crystals which have been selected are known to deform through different microscopic mechanisms. The corresponding data have already been published [8] but are reanalyzed here through different procedures.

More precisely, for a given material at given strain, a curve is plotted representing experimental data of \( \dot{\gamma}_{\text{pi}} \) as a function of \( \Delta \tau \). Then the following questions will be addressed:

i) How accurately does the hyperbolic sine relation fit this curve?

ii) How well can an extrapolation of this curve guide the experimentalist in the choice of \( \Delta \tau \) values. Indeed, the extrapolation starts from the first low \( \Delta \tau \) values, could indicate an approximate value of \( \tau^* \), and suggest a choice of \( \Delta \tau \) values larger than \( \tau^* \), thus saving time. In the result section, dotted blue curves refer to a fit considering all the data points, red ones refer to a few first points at low \( \Delta \tau \) values.

2. Experimental methods

2.1 Materials

The single crystals under investigation consist of Ni\(_3\)Al with 3at%Hf tested at 573K (0.35 of melting point temperature \( T_M \)), intrinsic Ge tested at 750K (0.62 \( T_M \)), Cu tested at 295K (0.22 \( T_M \)), Cu with 5at%Al and 7.5at%Al tested at 295K (0.22 \( T_M \)), referred to as Cu5Al and Cu7Al respectively. These crystals are known to deform according to different mechanisms. They were cut in the \([−123] \) single slip orientation.

2.2. Mechanical testing

Stresses \( \tau \), strains \( \gamma \) and strain rates \( \dot{\gamma} \) are resolved with respect to the primary slip system.

Details about compression tests, stress reduction experiments and computer programs designed for machine control and data acquisition can be found in [8]. Shear strain rates \( \dot{\gamma} \) were in the range of \( 10^{-4} \) s\(^{-1}\). Tests are performed at relatively low homologous temperatures, except for Ge.

3. Results

A typical curve \( \dot{\gamma}_{\text{pi}} (\Delta \tau) \) is presented in Fig. 1 for Ni\(_3\)Al deformed at 573 K. The blue dotted curve is a fit of relation (2) with all the data. Given the experimental uncertainty, the fit is considered to be satisfactory. It yields a value \( \tau^* = 75.7 \) MPa. The red curve is a fit of relation (2) with the 5 first data points. It is very close to the blue curve yielding a very similar value of \( \tau^* = 77.8 \) MPa.

Fig. 2 illustrates a similar attempt for Ge at 750K. The red curve is a fit considering the 4 first data points. It yields \( \tau^* = 12.6 \) MPa. The blue curve is very close to the red one and yields \( \tau^* = 12.8 \) MPa.

In these two cases, \( \tau^* \) could be obtained from about 5 dip tests (i.e. 5 specimens) only, at low \( \Delta \tau \) values and extrapolating with relation (2).

Fig. 3 shows a very different behavior for Cu at 295 K. There is a wide range of \( \Delta \tau \) values for which \( \dot{\gamma}_{\text{pi}} \) is close to zero and never negative. Fitting relation (2) with all data points (dotted blue curve) is still acceptable, yielding a \( \tau^* \) value of 5.4 MPa. However, when the 4 first data points are considered, a very different curve is obtained as well as a different value of \( \tau^* = 3.0 \) MPa.
Cu-5Al (Fig. 4) exhibits a behaviour similar to Cu. The fitting curves are superimposed at low $\Delta \tau$ values but differ for $\Delta \tau > 7$ MPa. They yield quite different values of $\tau^*$ (8.8 MPa for the whole set of data and 4.5 MPa for the 6 first points).

Cu-7Al (Fig. 5) exhibits a peculiar behaviour. Some negative values of $\dot{\gamma}_p$ are observed. The two fitted curves are very similar up to $\Delta \tau = 11$ MPa. They yield $\tau^* = 6.8$ MPa when the 7 first points are considered and 7.8 MPa otherwise.

**Figure 1.** Reduced strain rate $\dot{\gamma}_p$ as a function of the stress reduction $\Delta \tau$ for a Ni$_3$Al single crystal at 573 K and 620 MPa. Relation (2) fits all data points for the dotted blue curve, the 5 first ones for the red one. The corresponding values of $\tau^*$ are indicated.

**Figure 2.** Same as Fig. 1 for a Ge single crystal at 750 K and 24 MPa. The red curve considers the 4 first data points.

**Figure 3.** Same as Fig. 1 for a Cu single crystal at 295 K and 47 MPa. The red curve considers the 4 first data points.

**Figure 4.** Same as Fig. 1 for a Cu-5Al single crystal at 295 K and 15 MPa. The red curve considers the 6 first data points.

4. Discussion

Table 1 summarizes the results. The estimated values of $\tau^*$ refer successively to the dotted blue and red curves of the figures. A comparison of the curves on the figures shows that Ni$_3$Al and Ge behave
similarly according to relation (2). $\tau^*$ can be determined from the whole curve or the few first points. However, the slopes of the curves for $\dot{\gamma}_p = 0$ are different. Taking the derivative of (2) for $\Delta \tau = \tau^*$ yields a slope of $-AV/kT$, which has different values for the two crystals under different conditions. Cu and Cu-5Al exhibit no clear evidence of negative $\dot{\gamma}_p$ over a large range of $\Delta \tau$ values. The behaviour of Cu-7Al is rather similar. It is thought that the forest mechanism is operating in Cu and Cu-5Al so that the backward jumps of dislocations are unlikely thus excluding negative $\dot{\gamma}_p$ values. In this case $V$ depends on stress and therefore relation (2) is not justified.

![Graph showing Cu-7.5%Al, RT with $\tau^* = 6.8$ MPa and $\tau^* = 7.8$ MPa](image)

**Table 1.** Comparison of mechanical test data for the different materials.

| Sample  | T K  | $\tau$ (MPa) | $\tau^*$ (MPa) | $\dot{\gamma}_p^*$ (MPa) |
|---------|------|--------------|----------------|-------------------------|
| Ni$_3$Al | 573  | 620          | 75.7           | 77.8                    |
| Ge      | 750  | 24           | 12.8           | 12.6                    |
| Cu      | 295  | 47           | 5.4            | 3.0                     |
| Cu-5Al  | 295  | 15           | 8.8            | 4.5                     |
| Cu-7Al  | 295  | 30           | 7.8            | 6.8                     |

**Figure 5.** Same as Fig. 1 for a Cu-7Al single crystal at 295 K and 30 MPa. The red curve considers the 7 first data points.

5. Conclusions
The hyperbolic sine relation accounts for the dip test data in Ni$_3$Al (573 K, 620 MPa) and Ge (750 K, 24 MPa). It can be used safely to determine $\tau^*$ and save samples and experimental time. However, for Cu, Cu-5Al the forest mechanism seems to be rate controlling, negative values of $\dot{\gamma}_p$ predicted by Milicka's relation are not observed. Cu-7Al exhibits an intermediate behaviour.

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