

# Effect of strain on stored energy and recrystallization of straight-rolled silver

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## Abstract

Stored energy measurements were taken using a heat flux differential calorimeter, and the recrystallization kinetics were established for high purity and commercial silver straight rolled between 55% and 95%. The stored energy of deformation was found to increase almost linearly with the strain except for two well defined degrees of rolling. These “critical” strains exhibit a decrease in stored energy and an acceleration of recrystallization. There is some evidence that increasing the degree of deformation can lead to structure transitions which accelerate the recrystallization. This acceleration was identified as being a result of stimulated nucleation arising from the shear bands.

## 1. Introduction

In a metal, the energy  $E_s$  stored in the defects created during deformation represents the driving force of the reorganization of the lattice during the recrystallization. Determining this energy, through measurement of the energy released  $E_l$  during study of the recrystallization kinetics, enables us to understand better the nature of these defects [1]. Thus the few studies that have been devoted to the measurement of  $E_l$  as a function of rolling strain have shown [2–5] that the stored energy increases linearly with strain until, at high strains, a saturation value of this energy is reached. Only Lucci *et al.* [6] for copper and Salhi *et al.* [7] for silver have reported the existence of a decrease in the energy liberated  $E_l$  at a certain degree of rolling (63% for copper and 73% for silver). This article presents the results of a systematic study, carried out on pure silver, of the influence of the degree of rolling  $\eta$  on the energy released over a wider deformation range than that used by Salhi *et al.*, using a very sensitive calorimeter working in isothermal mode. An earlier study [8] was able to show that using this type of isothermal measurement under certain conditions,  $E_l$  could be assimilated to  $E_s$ .

The influence of purity on the “critical” strains was also studied using 99.99% pure silver.

## 2. Experimental method

A 99.999% pure Johnson Matthey silver ingot was rolled to a thickness  $l_0 = 4$  mm. After rolling, the

samples were obtained by cutting the sheet, and annealing was performed at 800 °C under a vacuum of  $10^{-5}$  Torr to provide an equiaxed grain structure. These samples of small grains were then deformed by straight rolling at room temperature to a thickness  $l$  to give  $\eta = 100(l_0 - l)/l_0$ .

The recrystallization was studied using a Calvet type [9] heat flux differential calorimeter including two thermoelectric cells mounted in opposition which constitute the laboratory and test cells. During recrystallization the heat effect is detected from the rolled sample in the laboratory cell while the test cell contains a sample of the same characteristics which was not deformed. The thermal output is measured after amplification by both analogical and digital means using a digital voltmeter coupled to a computer.

A thermogram, which is the recorded calorimetric curve  $\delta(t)$ , is a representation of the behaviour of the thermal phenomenon studied  $W(t)$ . When this process is as slow as recrystallization,  $W(t)$  can be linked to  $\delta(t)$  by Tian's equation [9]:

$$W(t) = \alpha \left[ \delta(t) + \tau \frac{d\delta(t)}{dt} \right]$$

where  $\alpha$  is a calibration factor and  $\tau$  the laboratory cell time constant.

The quantity of heat released at time  $t$  is determined from integration of the previous equation:

$$Q(t) = \int_0^t W(t) dt$$

The total quantity of heat released during the recrystallization is given as

$$Q_x = \int_0^x W(t) dt = E_1$$

It should be pointed out that the beginning of the thermogram must undergo a slight correction to take account of the fast endothermic effect ( $t \approx 1$  h) linked to an increase in sample temperature when reaching equilibrium from room temperature to the recrystallization temperature  $T_R$ . This correction is done from a blank curve obtained from the same sample after recrystallization. Note that, over the range of  $T_R$  used, the recrystallization is a slow exothermic effect ( $t \approx 20$  h), so the correction is generally negligible and does not disturb the kinetics studied [10].

From the values of  $Q(t)$  we have defined a variable  $F(t)$  such that

$$F(t) \frac{Q_x - Q(t)}{Q_x} = 1 - f(t)$$

where  $f(t)$  corresponds to the fraction of recrystallization at time  $t$ . The curves  $F(t)$  as a function of time give access to kinetic parameters such as  $t_{0.5}$  (times for which  $f(t) = 50\%$ ).

### 3. Experimental results

#### 3.1. Influence of the rolling strain on $E_1$

We varied  $\eta$  between 55% and 95% while adopting the recrystallization temperature which would give the greatest precision for the thermograms.

**High purity silver.** The results obtained for Johnson Matthey silver (99.999%) show, at a recrystallization temperature  $T_R = 57^\circ\text{C}$ , in the range of pronounced deformations not explored by Salhi *et al.* before, a new critical degree of reduction  $\eta_{2c} = 85\%$  (see Fig. 1(a)). The presence of  $\eta_{2c}$  was confirmed in other experiments carried out for higher reductions and a recrystallization temperature of  $48^\circ\text{C}$  (see Fig. 1(b)).

It should be pointed out in Fig. 1 that, for a given degree of deformation,  $E_1(T_R = 57^\circ\text{C}) > E_1(T_R = 48^\circ\text{C})$ . This variation of released energy is in fair agreement with our previous analysis concerning the effect of temperature on stored energy from isothermal calorimetric measurements [11].

**99.99% pure silver.** This metal was supplied by the Comptoir Lyon Aleman and Co. Figure 2 also indicates for this purity, at a recrystallization temperature  $T_R = 77^\circ\text{C}$ , the existence of two critical degrees of reduction. However there is a shift in  $\eta_{2c}$  which shows

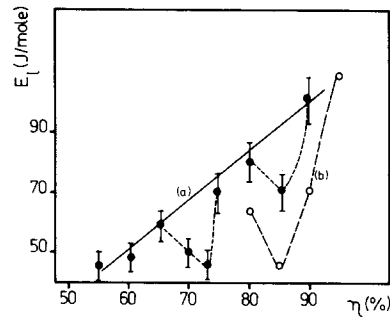


Fig. 1. Released energy as a function of strain for high purity silver (99.999%); (a)  $T_R = 57^\circ\text{C}$ , (b)  $T_R = 48^\circ\text{C}$ .

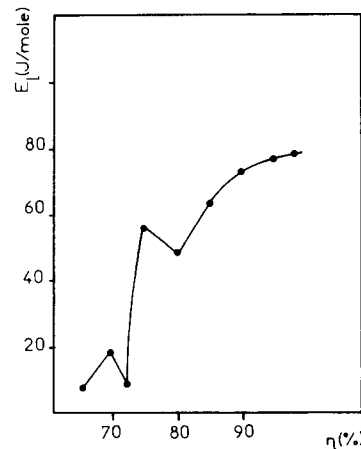


Fig. 2. Released energy as a function of strain for commercial silver (99.99%),  $T_R = 77^\circ\text{C}$ .

up for a slightly smaller degree of reduction (80%) than for the Johnson Matthey silver.

#### 3.2. Kinetics of recrystallization

The study of thermograms shows that for a critical degree of rolling, at the beginning of the recrystallization there is an athermal stage where no power  $W(t)$  is detected. Such a phenomenon is characteristic of an incubation stage corresponding to a phase of nucleation [10]. It is to be noted that this stage can no longer be

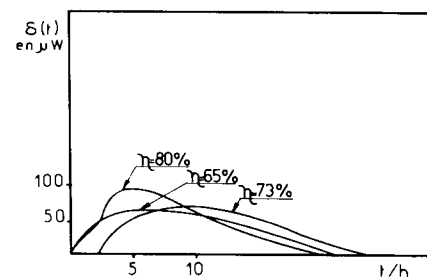


Fig. 3. Thermograms of recrystallization as a function of strain near  $\eta_{1c} = 73\%$ .

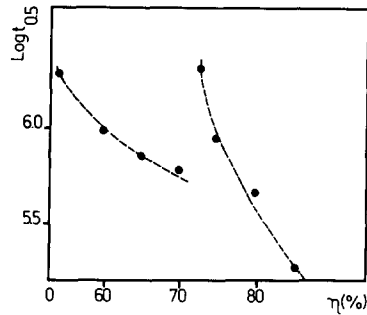


Fig. 4. Effect of strain on recrystallization kinetics.

observed for the other degrees  $\eta$  near  $\eta_c$  as is shown in Fig. 3 where the thermograms indicate a change of behaviour for each of three strains  $\eta = 65\%$ ,  $73\%$  and  $80\%$ .

The values of  $\ln t_{0.5}$  plotted as a function of strain show an acceleration of recrystallization for  $\eta > 73\%$  as indicated in Fig. 4.

#### 4. Discussion

Recrystallization, by definition, should involve the migration of high angle boundaries. Hence, except under special circumstances, a metal recrystallizes with changes in texture. Changes in microstructure are associated with these changes in texture. From the results accumulated [12] on the deformation and recrystallization textures, or microstructures, we can try to understand the evolution of deformed samples at increasing degrees of rolling. In particular, the relationships between the developing structure and texture in unidirectionally rolled brass which has the same textures of rolling as silver [13] have been the subject of many recent studies and are quite well understood: at  $\eta > 40\%$  large volumes of material deform by twinning and at about 60% reduction a texture transition occurs with a build-up of  $\{111\} \langle uvw \rangle$  components. This arises from coupled rotation of twins and matrix until twinning planes are parallel to the sheet surface producing two twin related  $\{111\} \langle 112 \rangle$  components [14]. Grains in which the laminar twin structure is close to this orientation form shear bands at about 70%. So these bands are indicative of macroscopic deformation processes which traverse many grains and contain severe local lattice rotations [15]. The multiplication and the progressive enlarging of these bands, when the degree of deformation increases, divide the laminar twin structure into rhomboidal prisms at 80% reduction [13]. Thus in a straight rolling, the structure which disappears is not slowly consumed by new grains nucleated elsewhere in the microstructure, as recently shown in

cross rolling [16], which allows us to envisage structure transitions in well defined degrees of deformation. In this connection our results show some salient points.

(i) In our study,  $\eta_{1c} = 73\%$  most probably corresponds to the appearance of shear bands for high purity silver [8]. Since a variety of previous works have established that localized shear bands contain copious recrystallization nuclei [17], it is of value to consider the influence of these nuclei relative to those occurring at other nucleation sites, such as grain boundaries, on the kinetics of recrystallization. Thus (a) the thermograms show clearly the nucleation and growth of new grains from the shear bands for  $\eta = 73\%$ , and (b) for high reduction, numerous shear bands are developed with large local misorientations. The growth of grains initiated at lower strains ( $\eta < 73\%$ ) at grain boundaries rapidly decreases and for higher deformations the overall recrystallization is dominated by the growth of new grains from shear bands which accelerate the kinetics.

(ii) Similarly,  $\eta_{2c} = 85\%$  agrees with the appearance of rhomboidal prisms in the structure of shear bands.

(iii) The decrease in released energy for the critical degrees of rolling can be correlated with the energy consumed by the structure transitions mentioned, or may reflect the heterogeneity of stored energy inherent in the deformed state, or textural differences. Note that the decrease in energy can also be linked to the possibility of fractures occurring at  $\eta_c$  inside the grains or at the grain boundaries [16].

(iv) The growth of the grains in high purity and commercial silver appears to be due to the movement of grain boundaries of different types, with different mobilities, in the various strain ranges studied.

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