Changes on dislocation energy and subgrain formation in Cu–16 at. % Al at T = 723 K and $\sigma = 39.2$ MPa according to the soft and hard region theory

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ABSTRACT. Using linear elasticity and the soft and hard region theory for internal stress we show that a mobile dislocation system may evolve from a disordered to a more organized structure by means of a highly dissipative process. This situation is in full agreement with the theory of self-organization in non-equilibrium irreversible systems. For the case of Cu-16 at. % Al at T = 723 K and $\sigma = 39.2$ MPa, our theory gives a quantitative explanation for the time evolution of the mobile dislocation density before the subgrain formation starts. It also provides such explanation for the evolution of the total dislocation density after the subgrain formation starts but annihilation events have not appeared yet in the dipolar structure of the subgrain walls.

RESUMEN. El uso de la teoría de las regiones blandas y duras para el esfuerzo interno junto con algunos elementos de teoría de elasticidad lineal permiten mostrar la capacidad que tiene el sistema de dislocaciones móviles (bajo la acción de un esfuerzo aplicado externo a la muestra) para evolucionar de una estructura desordenada a otra más organizada a través de procesos muy disipativos. Esto está en completo acuerdo con la teoría sobre la autoorganización que exhiben los sistemas irreversibles fuera de equilibrio. Para el caso de Cu–16 % at. Al a T = 723 K y $\sigma = 39.2$ MPa, nuestra teoría aporta una explicación cuantitativa para la evolución temporal de la densidad de dislocaciones móviles antes de que dé comienzo la formación de subgranos de dislocaciones. Además, nuestra teoría describe la evolución de la densidad total de dislocaciones para el período que va desde el comienzo de la formación de los subgranos hasta antes de que ocurran eventos de aniquilación en la estructura dipolar de las paredes de subgrano.

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1. INTRODUCTION

From a theoretical point of view, the process of subgrain formation during plastic deformation in metals has been a subject of interest from a long time ago. (See the pioneer works due to Kuhlmann-Wilsdorf [1] and Holt [2].) Recently, this problem has received increasing attention [3–11]. The analysis of experimental past research leads to the conclusion that dislocation subgrain formation in work-hardened crystalline material occurs when dislocations assemble into low energy configurations (see Kuhlmann-Wilsdorf *et al.* [12]). Most of the theoretical analyses about the process of dislocation subgrain formation have been performed within a reversible thermodynamical scheme (see for instance Kuhlmann-Wilsdorf and Van Der Merwe [13]); however the plastic deformation of crystals is an irreversible process [14].

According to the most developed reversible thermodynamical model, the mesh length theory, due to Kuhlmann-Wilsdorf [15], low energy dislocation structure, LEDS, must be stress screened, *i.e.*, presumably the range of the stress field of the average dislocation in a LEDS extends more or less only to the nearest neighbor dislocation. By contrast, the central result of an X-ray study on Copper single crystals, due to Mughrabi *et al.* [16, 17], is that substantial long-range internal stresses prevail in deformed crystals containing a well developed subgrain structure. According to Mughrabi this finding is somewhat unusual, since there exists a wide belief that long-range internal stresses should be insignificant in dislocation cell structures or subgrain structures, both being considered to be low energy dislocation arrays [15].

According to Nabarro [18] a model for internal stress, σ_i , developed initially by Nix and Ilschner [19] and later on developed with considerable detail by Mughrabi [20,21], provides an adequate description of the internal stress distribution revealed by the X-ray analysis [16] on f.c.c. crystals undergoing plastic deformation.

The soft and hard region theory, SHRT, due to Mughrabi [20, 21], has been shown to be very useful for interpreting numerous phenomena in the plastic behavior of metals and solid solution alloys [11, 22-33]. In particular, a simplified version of SHRT [11] has been used in the microscopic theory of the Bailey-Orowan equation; the corresponding irreversible thermodynamical analysis yields the following two main results: 1) It gives a qualitative answer to the question of what are the basic mechanisms which lead to the formation of dislocation subgrains. Also, they establish that at steady state the dissipative subgrain wall structure remains statistically invariant because there is a constant flow of external mechanical energy given by $\dot{\rho}_{\rm m}^+ \bar{u}_{\rm loop}$, which restores in a dynamical way the power $\dot{\rho}_{\rm w}^- \bar{u}_{\rm loop}$ dissipated by annihilation events. (Here, $\dot{\rho}_{\rm m}^+$ and $\dot{\rho}_{\rm w}^-$ are respectively the creation rate of mobile dislocation density and the annihilation rate of dislocation density at the center of the dipolar structure of subgrain walls, and finally \bar{u}_{loop} is the mean value of the total energy of dislocation loops per unit length.) 2) This analysis leads to a quantitative expression which relates the subgrain diameter, d_{sg} , with the applied stress σ , for steady state power law creep. $d_{sg} = K\mu b/\sigma$, where μ and b have their usual meanings. For the first time the value of the phenomenological constant K is obtained here as a function of basic parameters of plastic deformation and can be predicted. The percentage difference between theoretical and experimental values for typical metals is 10%. Neither of these two problems have been answered unambiguously by the mesh length theory [34]. Also the same simplified version of the SHRT gives a full description of the time evolution of the internal stress during sigmoidal transient creep in Cu-16 at. % Al; including the creep stage where no subgrain structures have formed yet [33].

In this work, a study about the subgrain formation process will be made by looking its origin in terms of a free energy. Our analysis starts from the simplified version of the

soft and hard region theory mentioned before [11, 33] and leads to an expression for the mean value of the total energy, $\bar{u}_{\rm T}$, of dislocation per unit length. Following a Seeger [35] suggestion, and with the use of our expression, we are able to show the capability of the mobile dislocation system to evolve from a disordered (no subgrain exists) to a more organized structure (subgrain formation under the action of an externally applied stress, σ) by means of a highly dissipative processes. According to our model, for the case of Cu-16 at. % Al samples [36] at T = 723 K and $\sigma = 39.2$ MPa during the time where the subgrain structures begin to appear, twenty five percent of the total stored energy by mobile dislocations is dissipated by a sudden decrease of the mean energy of dislocations per unit length without any annihilation events of dislocations at the first stage of subgrain formation. This last situation is in full agreement with the theory of self organization in non-equilibrium irreversible systems (see Prigogine and coworkers [37, 38]). In other words, starting from a long-range internal stress theory and using some elements of irreversible thermodynamics, we are able to provide a description of the subgrain formation which is in agreement with the low energy dislocation structure hypothesis.

2. The total energy of dislocations during creep

When a crystal is deformed plastically, the stored energy by dislocations is composed of two parts [13]. The first one is the dislocation line energy of all the dislocations per unit volume. The second part is the energy of overall dislocations cell stresses. In other words, one could say that the first part is the self energy of all dislocations stored in the crystal and the second part is the average interaction energy between all dislocations. For simplicity, we have explicitly avoided the consideration of closed dislocations of the Frank type which are due to the confinement of vacant sites. Therefore, before we engage in the theoretical development, a brief word on the self energy of dislocations is called for.

The energy u per unit length of a straight dislocation line is, to a first approximation [39],

$$u = \frac{Gb^2 f(\nu)}{4\pi} \ln\left(\frac{x_t}{r_0}\right),\tag{1}$$

where G is the shear modulus, b is the magnitude of the Burgers vector, $f(\nu)$ is a function of Poisson's ratio ν . $f(\nu)$ is $\frac{1}{(1-\nu)}$ and unity for an edge dislocation and a screw dislocation respectively. And for an average dislocation population it equals about $(1 - \nu/2)/(1 - \nu)$. x_t is a correlation distance characterizing the more distant dislocation interaction in the linear elastic approximation (see Kocks, Argon and Ashby [14]) and $r_0 = 5b$ [33, 40]. Then the mean self energy of dislocations, U, per unit volume, may be expressed as

$$U_{j} = \rho_{j} \left[\frac{1.15}{8\pi} \frac{(2-\nu)}{(1-\nu)} G b^{2} \ln\left(\frac{x_{t}}{r_{0}}\right) \right]$$
(2)

$$\equiv \rho_j u_{\text{mean}}(x_t),\tag{3}$$

where ρ_j is the mobile dislocation density for j = m, and it is the subgrain dislocation density ρ_w for $j = \omega$. The factor (1.15) appears due to the contribution of the dislocation

core [41]. We make note that in the present state of knowledge of the stored energy of dislocations in plastically deformed materials, expressions very similar to Eqs. (2) and (3) have been used to describe experimental [42–45] and theoretical aspects [13, 15, 21, 40, 46] of the problem.

According to Hansen and Kuhlmann-Wilsdorf [39], in their excellent review on a subject related to the self energy of dislocations, usually the value of x_t in Eq. (1) has been taken to be the average dislocation spacing, *i.e.*,

$$x_t \sim 1/\rho^{1/2},$$
 (4)

where ρ is the local dislocation density. This choice of x_t is valid provided that the dislocation structure is "stress screened", meaning that the dislocations are arranged so as to screen each other's stress field mutually. This assumption, [Eq. (4)], which is in agreement with the mesh length theory [15] is in contradiction with the X-ray study on Copper due to Mughrabi *et al.* [16,17], where it was shown that substantial long-range internal stresses prevail in deformed crystals containing a well developed subgrain structure.

How, then, can one choose x_t so that it is on the one hand consistent with the experimental evidence provided by Mughrabi *et al.* [16, 17] and also accounts for the fact that x_t is a correlation distance characterizing the more distant dislocation interaction (see Kocks *et al.* [14])?

A possible route to answer this question has been given in the previously mentioned review paper [41]. They point out that "measurements of stored energy by calorimetry have been made in large numbers and with increasing accuracy. These are helpful in supplying additional data which must be explained through theory. However, in this, moderate variations in R ($R = x_t$ in our notation), cannot be derived to account for discrepancies between measured and calculated values, for the reason that the logarithmic term in Eq. (1) is lowly varying function". Another possibility, however, is to look for sources of data different from calorimetry that could shed some light on a criterion to specify x_t . Here we will follow this second approach. According to Kocks, Argon and Ashby [14] x_t is a correlation distance characterizing the more distant dislocation interaction in the linear elastic approximation. In other words, x_t is the maximum distance within which a discontinuous (non linear) change in the crystalline lattice orientation takes place. With this consideration, x_t equals the grain size for samples in which subgrains are not formed yet by plastic deformation, in agreement with previous results [47, 48]. For the case where subgrain have been formed by plastic deformation x_t will be equal to the subgrain or cell size [24, 40].

Once we have digressed on the self-energy of dislocations, we now turn to the second contribution to the dislocation stored energy during creep. The interaction energy of all dislocations existing in a sample deformed in a creep test will be calculated by using the simplified version of the soft and hard region theory [11,33]. According to this model, the long-range internal stress σ_i can be written as

$$\sigma_{\rm i} = \alpha M G b (\sqrt{\rho_{\rm m}} + \sqrt{\rho_{\rm w}}) \tag{5}$$

$$\equiv \sigma_{\rm im} + \sigma_{\rm i\omega},\tag{6}$$

where M is the Taylor factor relating the macroscopic tensile stress to the shear stress in active slip systems, and α is a quantity which characterizes the interaction of dislocations and depends on the geometrical arrangement of the dislocation structure and on the character of dislocations as a whole [49]; as a first approximation it remains constant during deformation [50]. $\sigma_{\rm im}$ and $\sigma_{i\omega}$ are used respectively for the internal stress due to mobile dislocations and for the internal stress due to subgrain wall dislocations. It should be noted that Most engineering structures are influenced by complex multiaxial stresses that arise from loading, geometry, and/or material inhomogeneity. However, laboratory testing in the creep regime predominantly uses simple specimens that are subjected to uniaxial states of stress [51].

The stored strain energy U_{σ_i} per unit volume due to an axial stress σ_i [41] is given by $U_{\sigma_i} = (\sigma_i)^2/(2E)$, where the Young modulus E is related with the shear modulus G by the standard relation $G = E/[2(1 + \nu)]$. Then by using Eq. (5) in the previous expression for U_{σ_i} leads to

$$U_{\sigma_{\rm i}} = \frac{(\alpha M)^2}{4(1+\nu)} Gb^2(\rho_{\rm m} + \rho_{\rm w} + 2\sqrt{\rho_{\rm m}}\sqrt{\rho_{\rm w}}),\tag{7}$$

where the first and second terms are respectively the stored strain energy per unit volume due to elastic deformation arising from the stress field of $\rho_{\rm m}$ and $\rho_{\rm w}$. The third term is an interaction energy between mobile and subgrain wall dislocation densities. In this equation it is clear that for a constant value of the total dislocation density, U_{σ_i} is bigger if the material has subgrains than if all dislocations remain mobile.

Finally, the total stored energy, $U_{\text{Ts.g.}}$, per unit volume due to dislocations is equal to the sum of the volumetric density, U_{m} , of the self energy of mobile dislocations, the corresponding density, U_{W} due to subgrain wall dislocations and the stored strain energy U_{σ_i} per unit volume due to the internal stress σ_i . That is,

$$U_{\mathrm{Ts.g.}} = U_{\mathrm{m}} + U_{\mathrm{W}} + U_{\sigma_{\mathrm{i}}},\tag{8}$$

where $U_{\rm m}$ and $U_{\rm W}$ are given by Eq. (1) and $U_{\sigma_{\rm i}}$ by Eq. (7).

With the use of the definition of the total dislocation density $\rho_{\rm T} = \rho_{\rm m} + \rho_{\rm w}$, and substituting Eqs. (1) and (7) into Eq. (8), an expression for the mean value of the total energy of dislocations, $\bar{u}_{\rm T}$, per unit length during deformation in power-law creep can be obtained, namely,

$$\bar{u}_{\text{Ts.g.}} \equiv \frac{U_{\text{Ts.g.}}}{\rho_{\text{T}}} = u_{\text{mean}}(x_t) \left[1 + \frac{(\alpha M)^2}{4(1+\nu)} \frac{Gb^2}{u_{\text{mean}}(x_t)} \left(1 + \frac{2\sqrt{\rho_{\text{m}}}\sqrt{\rho_{\text{w}}}}{\rho_{\text{T}}} \right) \right].$$
(9)

The mean value for the energy of dislocation, $u_{\text{mean}}(x_t)$, per unit length can be experimentally determined [24, 33, 45] or calculated by using some models [52, 53]. Equation (9) is the main result of this section.

3. NUMERICAL CALCULATIONS AND COMPARISON WITH EXPERIMENTAL DATA

The merits of Eq. (9) will now be assessed on two different fronts. On the one hand, by considering typical numerical values of the physical parameters involved, we will perform some calculations to examine the evolution of a system originally formed by mobile dislocations into a more organized one in which mobile dislocations as well as dislocations forming subgrain walls occur. On the other hand, we will use an approximate form of Eq. (9) together with a time evolution equation for $\dot{\rho}_{\rm m}^+$ in which $\bar{u}_{\rm T}$ is involved [40], to make a comparison with experimental data of $\rho_{\rm m}$ and $\rho_{\rm T}$ vs. t for Cu-16 at. % Al [36].

3.1. General calculations and the low energy dislocation structure hypothesis

Concerning the first aspect, if no subgrain walls have formed, x_t is equal to the crystal size for single crystals or to the grain size for polycrystalline materials. Then for this case Eq. (9) appears like,

$$\bar{u}_{\rm h} = u_{\rm mh}(x_t) \left[1 + \frac{(\alpha M)^2}{4(1+\nu)} \frac{Gb^2}{u_{\rm mh}(x_t)} \right],$$
(10)

where $u_{\rm mh}(x_t)$ is the mean energy for dislocations when an homogeneous distribution exists. On the other hand, when dislocation subgrains have been formed $\rho_{\rm m}/\rho_{\rm T} \cong$ 0.10 [36,54], and then by using Eq. (9), an expression to evaluate the fraction of elastic energy stored during the early process of subgrain formation (where no annihilation events occur) can be obtained, namely

$$\frac{u_{\rm Ts.g.}}{u_{\rm th}} = \left(\frac{u_{\rm m.s.g.}}{u_{\rm m.h.}}\right) \left(\frac{1 + \frac{1.6\,AGb^2}{u_{\rm m.s.g.}}}{1 + \frac{AGb^2}{u_{\rm m.h.}}}\right),\tag{11}$$

where $A \equiv (\alpha M)^2 / [4(1 + \nu)].$

By considering typical numerical values of the physical parameters involved, a calculation of the ratio $u_{\text{T.s.g.}}/u_{\text{th}}$ is presented in Fig. 1. In order to make such calculations the following values were used, $u_{\text{Th}} = 2Gb^2$. M = 3.1 for polycrystals [55], a typical value for the Poisson's ratio $\nu = 0.32$, and α values ranging from 0.017 to 2.5 were taken [56]. All the curves displayed in Fig. 1, show that a mobile dislocation system may evolve from a disordered to a more organized structure by means of a highly dissipative process. This situation is in full agreement with the theory of self organization in non-equilibrium irreversible systems.

In particular, from these curves it is clear that subgrain structures are energetically more stable for small α ($\alpha < 0.65$) than subgrain structures formed for materials with bigger interaction constant between dislocations ($1.5 < \alpha < 2.5$). We make note that our theoretical analysis is also in agreement with the low energy dislocation structure hypothesis. This hypothesis states that dislocations and subgrains are expected to approach the configuration of lowest possible stored strain energy at constant total dislocation line length. Therefore, according with our model the values of α which ensure the fulfilment



FIGURE 1. The ratio $u_{\text{Ts.g.}}/u_{\text{Th}}$ as a function of the parameter $A = (\alpha M)^2/[4(1+\nu)]$. This fraction of stored energy when subgrains appear is presented as different curves labeled by the value of the ratio $r_{\text{iso}} = u_{\text{iso s.g.}}/u_{\text{iso h}}$, where $u_{\text{iso s.g.}}$ is the mean energy for isolated dislocations when subgrains begins to appear and $u_{\text{iso h}}$ is the mean energy per unit length for isolated dislocation when an homogeneous dislocation distribution already exist.

of the LEDS hypothesis are the lowest values compatibles with theoretical values or experimental data.

3.2. Stored energy by dislocations in Cu

In the following paragraphs, an specific calculation for Copper will be done. If no subgrain walls have formed, x_i is equal to the crystal size for single crystals or to the grain size for polycrystalline materials. In the former case $x_t \sim 5 \times 10^{-3}$ m [57]. On the other hand, when dislocations cells or subgrains are present, our assumption implies that x_t is the cell or subgrain size, typically $x_t \sim 4 \times 10^{-5}$ m [58]. Further, for polycrystals with $\nu = 0.32$, $b = 2.5 \times 10^{-10}$ m, and M = 3.1 [59]. The case of α in the frame of our model it is solved as follows. According to Lavrantev [56], an analysis of the available data and a comparison of α values obtained by different authors on single crystals of the same materials shows marked differences. For instance, α values for copper range between 0.15 to 0.65. Also, Lavrantev indicates that the experimental estimates of α do not give preference to any of the review models for work-hardening mechanisms. Since, nevertheless, the Taylor model [60] or Taylor lattice [61] consisting of parallel rows of like edge dislocations whose sign changes from row to row, has been used to develop more sophisticated work-hardening models (and is considered to be a low energy dislocation structure [15]). Here, based on our theoretical analysis previously presented, we take the lowest values of α compatibles with the theoretical and experimental data previously mentioned ($\alpha = 0.16$). The condition that during plastic deformation no subgrains have yet been formed, the mean energy for an isolated dislocation $u_{isoh}(x_{tc.s.} = 5 \times 10^{-3} \text{ m}) = 1.72 \, Gb^2$ and from Eq. (9), one has $u_{TCS} = 1.78 \, Gb^2$, *i.e.*, the total energy of mobile dislocations per unit length remains constant. On the other hand, when dislocation subgrains have been formed, with a typical initial diameter around $4 \times 10^{-5} \text{ m} (\sim x_t)$, the mean energy of an isolated dislocation is then $u_{iso s.g.}(x_{ts.g.} = 4 \times 10^{-5} \text{ m}) = 1.17 \, Gb^2$, and it follows from Eq. (9) that $u_{Ts.g.} = 1.25 \, Gb^2$, where we have taken $\frac{\rho_m}{\rho_T} \cong 0.10$ as determined from experimental data once subgrains may be detected [36, 54]. A comparison between u_{TCS} and $u_{Ts.g.}$ indicates that even without dislocation annihilation events occurring in this process of subgrain formation, a great deal of stored elastic energy is dissipated (29.8%).

In order to be in a position to indicate how such dissipation could be detected through calorimeter measurements, we will now present a quantitative estimate of this energy which rests on a theoretical result [62] according to which $\rho_{\rm m} = 3 \times 10^9$ cm⁻² when the subgrains structure begins to develop. In the case of Copper, $G = 46 \times 10^3$ MPa, so that the energy dissipated through the initial process of subgrain formation would be 4.6×10^{-5} J/mm³, *i.e.*, about a 3% of the greatest value of the stored energy reported by Rönnpagel and Schwink [44] for Cu in tensile deformation at room temperature and constant speed of deformation. Such a value of the dissipated energy must also show as a sudden increase in temperature, very well localized in time, followed by a progressive cooling until the system reaches the temperature it had before the formation of subgrains started. This temperature behavior, which incidentally is not predicted by the mesh length theory [15], should of course be easier to measure than the dissipated energy profile due to experimental limitations [44].

3.3. Stored energy by dislocations in Cu-16 at. % Al

Concerning the second aspect, a comparison between predictions of our model for the total dislocation energy and experimental data for $\rho_{\rm m}$ and $\rho_{\rm T}$ vs. t for Cu-16 at. % Al [36] will be made. To our knowledge, this is the only set of experimental data in which all parameters involved in the model are available. Here we follow the validation criterion suggested by Schoeck [63]: "Any physical model for $\frac{d\rho}{dt}$ can be checked independently at least in principle, since an integration should give the total dislocation density if no mutual annihilation takes place". Our analysis about the experimental data of $\rho_{\rm m}$ and $\rho_{\rm T}$ on Cu-16 at. % Al is restricted to the deformation stage where we consider that annihilation events of dislocations have not occurred yet.

Before we start our analysis, let us a briefly review the pertinent data on Cu-16 at. % Al. It should be noted that the alloy Cu-16 at. % Al at 723 K is above $0.5 T_{\rm m}$ (with $T_{\rm m}$



the melting temperature), so recrystallization processes might be expected. However as first pointed out by Hardwick, Sellers and Tegart [64], any process that lowers lattice strain energy as subgrain formation inhibits recrystallization. In the case of Hasegawa et



FIGURE 2. Various parameters as a function of creep time (Cu-16 at. % Al, 723 K, 29.2 MPa taken from Ref. [36]): (a) creep strain ε and creep rate $\dot{\varepsilon}$; (b) total dislocation density $\rho_{\rm T}$ and ratio $L_{\rm cell}/L_{\rm TOTAL}$ of length of dislocations within cells to total length of dislocations; (c) internal stress level.

al. [36] data, no experimental evidence of recrystallization can be ascertained. In Fig. 2 the experimental creep data on Cu-16 at. % Al, due to Hasegawa et al. [36] for the strain rate, $\dot{\varepsilon}$, dislocation density and internal stress level $\frac{\sigma_i}{\sigma}$ are shown at 723 K and $\sigma = 39.2$ MPa. Here cell dislocations are taken as being mobile, and the total dislocation density $\rho_{\rm T}$ minus mobile dislocation density ρ_m yields the subgrain dislocation density ρ_w . In a recent work [33] by using a soft and hard region theory for σ_i , it has been shown that the experimental value of σ_i for a sigmoidal creep curve in Cu-16 at. % Al, can only be explained by considering the contribution to σ_i arising from subgrain wall dislocations, as well as from dislocations that do not belong to the subgrain walls. In this same work by using theoretical calculations on σ_i the authors arrive at the following conclusion: the determination of σ_i based on the stress dip test may be too perturbative for the sample when it such a procedure is applied at the stage of deformation for which subgrain wall of dislocations have not formed yet. The authors were able to explain this perturbation in a quantitative way. Their theoretical prediction for σ_i before this perturbation is shown in Fig. 3 by starred points. The solid line represents the experimental data for σ_i as obtained by Hasegawa et al. with the use of a dip test technique [36]. The expression for σ_i used in their analysis [33], was $\sigma_i = \alpha' Gb(\sqrt{\rho_m} + \sqrt{\rho_w})$, which is not the standard way used to



FIGURE 3. Comparison between prediction for σ_i before perturbation during the dip test procedure and the experimental creep data on Cu-16 at. % Al as presented by Hasegawa *et al.* [36]. The difference between solid line and starred points has been quantitatively explained elsewere [33].

express axial internal stresses (see for instance Eq. (5) or the review due to Orlová [49]). If we take into account that $\alpha' = \alpha M$, where M = 3.1 for a polycrystalline material as mentioned before, then $\alpha = 0.145$ for this alloy; this value is very close to the value for α given by the Taylor model ($\alpha = 0.16$) [60]. By using $\alpha' = \alpha M$ in the semi-empirical expressions due to Montemayor-Aldrete *et al.* [33], it is easy to show that the mean value of the total energy, $u_{\rm TCS}$, of dislocations per unit length (for the case were subgrains have not formed yet) has a corrected empirical value given by

$$u_{\rm TCS}(x_t = 3 \times 10^{-4} \text{ m}) = 3.95 \, Gb^2$$
 (12)

with the rest of predictions for Cu-16 at. % Al as the same as before.

in what follows, by using Eqs. (1), (9) and (11) a semiempirical determination of the total energy of dislocations, $u_{\text{Ts.g.}}$, per unit length for the case when subgrains already exist will be made. In order to do that we require to calculate the self energy per unit length of isolated dislocation loops $u_{\text{iso}\,\text{h}}(x_{\text{tc.s.}})$ for the case where subgrains have not formed yet ($\rho_{\rm w} = 0$). For this case, Eq. (9) with $\nu = 0.326$ [33], M = 3.1 for polycrystalline material [59], grain diameter $d_{\text{c.s.}} = x_t = 3 \times 10^{-4}$ m for Cu-16 at. % Al [36], $\alpha = 0.145$ as mentioned before, then

$$u_{\rm iso\,h}(x_{\rm tc.s.} = 3 \times 10^{-4} \text{ m}) = 3.91 \, Gb^2.$$
 (13)

On the other hand, for the case when dislocation subgrain have been formed by plastic deformation in Cu-16 at. % Al, we take a mean diameter $d_{\rm s.g.} = 1.3 \times 10^{-5}$ m, where $x_{\rm ts.g.} = d_{\rm s.g.}$ was calculated by using the phenomenological expression $d_{\rm s.g.} = KGb/\sigma$. With K as 39 for copper (see Raj and Pharr [65]), $\sigma = 39.2$ MPa [36], $G = 5.16 \times 10^4$ MPa and $b = 2.56 \times 10^{-10}$ m [33]. Then according to Eq. (1), it is clear that the following relationship must be fulfilled:

$$\frac{u_{\rm iso\,s.g.}(x_{\rm t\,s.g.} = 1.3 \times 10^{-5} \text{ m})}{u_{\rm iso\,h}(x_{\rm t\,c.s.} = 3 \times 10^{-4} \text{ m})} = 0.746,\tag{14}$$

where $u_{iso s.g.}(x_{t s.g.} = 1.3 \times 10^{-5} \text{ m})$ is the mean self-energy per unit length of isolated dislocation when subgrains, with an average diameter $d_{s.g.} = 1.3 \times 10^{-5}$ m exist. Therefore, in this case u_{iso} is given by

$$u_{\rm iso\,s.g.}(x_{\rm t\,s.g.} = 1.3 \times 10^{-5} \text{ m}) = 2.92 \, Gb^2.$$
 (15)

With this value, using Eq. (9) and considering, as before that in an approximate way $\rho_{\rm m}/\rho_{\rm T} \sim 0.10$ [36, 54], we finally obtain a mean value for the total energy $u_{\rm Ts.g.}$ for the case when subgrains have been formed in Cu-16 at. % Al,

$$u_{\text{T s.g.}}(x_t = 1.3 \times 10^{-5} \text{ m}) = 2.98 \, Gb^2.$$
 (16)

In Fig. 4 we display the behavior of the mean total energy of dislocations per unit length as a function of creep time, according to Eqs. (11) and (16).

From a comparison between Eq. (16) and Eq. (15) it is clear that the stored elastic strain energy due to the long-range internal stress, σ_i , represents only a small fraction, about 2%, of the mean self-energy of the dislocations. Similar results has been obtained previously on Copper by using the soft and hard region theory [24].

3.4. Evolution on dislocation density on Cu-16 at. % Al

At this point, with the knowledge of the values of the total dislocation energy per unit length for Cu-16 at. % Al, we can proceed to calculate the evolution on dislocation density over a period of time about 270 minutes after the starting of the creep test. Our calculations will be assessed on two parts: On the one hand, by calculating the mobile dislocation density versus time for times lower than the corresponding to the inflection point in the sigmoidal creep curve (where annihilation events of dislocation have not occurred yet [33]). On the other hand, for times larger than the one associated to the inflection point, we will evaluate the total dislocation density for a period of time where we consider that annihilation events of dislocations have not occurred yet. In both cases a comparison with experimental data will be made.

In order to do that we need a mechanistic model to describe the creation rate of mobile dislocations.



FIGURE 4. The mean value of the total energy, $u_{\rm T}$, of dislocations per unit length as a function of creep time for Cu-16 at. % Al 723 K, 39.2 MPa (as calculated by using Eq. (9) and $\rho_{\rm m}$ and $\rho_{\rm w}$ from Fig. 2b). The value of $u_{\rm T\,c.s.}$ is constant for the case of isolated dislocations, and for the case where subgrains exist the value of $u_{\rm T\,s.g.}$, which decreases monotonically with time does not change with time in more than 0.1% from its initial value.

According to Montemayor-Aldrete *et al.* [11, 33, 40, 66], the creation rate $\dot{\rho}_{\rm m}^+$ of mobile dislocations is given by

$$\dot{\rho}_{\rm m}^{+} = \frac{\phi_M \sigma \dot{\varepsilon}}{\bar{u}_{\rm T}},\tag{17}$$

where $\bar{u}_{\rm T}$ is the mean value of the total energy of dislocations per unit length and $\phi_M \equiv 1/M$.

Now we can calculate $\rho_{\rm m}$ vs. t, for the stage of deformation previous to the inflection point in the sigmoidal creep curve. If annihilation events of dislocations do not occur in a certain period of time $(t - t_0)$ then in such period of time $\frac{d\rho_{\rm m}}{dt} = \dot{\rho}_{\rm m}^+$, and then

$$\rho_{\rm m}(t) = \rho_{\rm m}(t_0) + \int_{t_0}^t \dot{\rho}_{\rm m}^+ dt, \qquad (18)$$

where $\rho_{\rm m}(t_0)$ is the density of mobile dislocations at the beginning of the creep test.

By using Eq. (12) and the data for strain rate $\dot{\epsilon}$ (see Fig. 1), $\rho_{\rm m}$ versus t is easily calculated. The theoretical curve and the experimental one are indistinguishable. To illustrate

this point, the value of $\rho_{\rm m}$ at the inflection point will be calculated. The total plastic deformation at the inflection point according to Fig. 2 is $(\varepsilon_p)_{\rm i.p.} = 7.8 \times 10^{-3}$ [33, 60]. $\sigma = 39.2$ MPa, M, G, and b are taken as before, and by using Eq. (11) the total dislocation energy $u_{\rm TC.S.} = 1.34 \times 10^{-10}$ MPa cm². Here $\rho_{\rm m}(t_0) = 5 \times 10^6$ cm⁻² (see Hasegawa et al. [36]). Therefore by using Eq. (18) we obtain

$$(\rho_{\rm m\,theo})_{\rm i.p.} = 7.41 \times 10^8 \,\,{\rm cm}^{-2},$$
(19)

with $(7.6 \pm 0.4) \times 10^8$ cm⁻² as the experimental value reported by Hasegawa *et al.* (Fig. 1).

On the other hand, during the lapse of time between the associated time to the inflection point in the creep curve and 270 minutes, the sample plastically deforms 4.5×10^{-3} (see Fig. 2), the total dislocation energy when subgrains already exist is $u_{\text{Ts.g.}} = 1.01 \times 10^{-10}$ MPA cm² [see Eq. (16)]. Then if we consider that no annihilation events take place between 164 minutes and 270 minutes, the following equation could be used to describe the value of ρ_{T} at 270 minutes:

$$\rho_{\rm T}(t = 270 \text{ min}) = (\rho_{\rm m\,theo})_{\rm i.p.} + \frac{\sigma \times 4.5 \times 10^{-3}}{3.1 \ \bar{u}_{\rm T\,s.g.}}.$$

Using the corresponding values on this equation a value for $\rho_{\rm T}$ it is obtained,

$$\rho_{\rm T\,theo}(t = 270 \,\,{\rm min}) = 1.30 \times 10^9 \,\,{\rm cm}^{-2},$$
(20)

with $(1.32 \pm 0.8) \times 10^9$ cm⁻² as the experimental value for $\rho_{\rm T}$ reported by Hasegawa *et al.* (see Fig. 2).

The comparison between theoretical and experimental results for $\rho_{\rm m}$ and $\rho_{\rm T}$ clearly indicates a good agreement. Then according to our model, based on the soft and hard region theory, during the lapse of time where subgrain begins to form in Cu-16 at. % Al at 723 K and $\sigma = 39.2$ MPa twenty five percent of the stored energy of mobile dislocations is dissipated in the sample by highly cooperative process without annihilation of dislocations.

4. DISCUSSION

Here we discuss some fundamental aspects of the experimental work on the stored energy in copper single crystals as reported by Rönnpagel and Schwink [44] and later analyzed by Ungar, Mughrabi, Rönnpagel and Wilkens [24]. Also some new experimental work on Cu-16 at. % Al or some Alkaline Halides is suggested in order that a prediction made by our theory may be tested in a definitive way.

The soft and hard region theory, SHRT, has been used previously [24] to explain in some way the elastic stored energy per unit volume, U_{stor} , in deformed copper single crystals. The value for x_t used by Ungar *et al.* in order to explain the calorimetry results is the cell size, but the U_{stor} experimental results are consistently larger than the ones that can be accounted for by dislocation line energy alone, by a factor of 2 to 2.5. Also, because the stored elastic strain energy due to the long-range internal stresses is about 2% of the

measured stored energy values, they consider that this discrepancy can be slightly reduced but cannot be resolved by including such contribution. Ungar *et al.* [24] consider that the main reason for the high values of U_{stor} appears to be the storage of some kind of lattice defects which are produced during the course of plastic deformation but which are neither visible in a normal, bright-field TEM micrograph. Weak-beam TEM observations due to Ungar *et al.* indicate the presence of small defect clusters (presumably dislocation loops, estimated diameter: 3 nm) in a mean volume density larger that 10^{22} m⁻³. According to Ungar *et al.* to clarify these questions further investigation is necessary.

Here an analysis on the Ungar *et al.* [24] and Rönnpagel *et al.* data [44] will be made in order to show that SHRT solves the problem mentioned above when it is used together with an analysis which takes into account the creation of vacancies and other point defects during plastic deformation.

According to Rönnpagel *et al.* [44] for all hardening theories in spite of the different models used, the stored energy by dislocations is proportional to the squared of shear stress, τ^2 , and also the stored energy by creation of point defects is proportional to τ^3 (see Saada [67]). In Fig. 11 of the Rönnpagel paper it is clear that for $\tau = 60$ MPa, the fraction of U_{stor} due to dislocations is 4.69 J/mol, and the fraction due to storage of point defects is 5.97 J/mol. If we take [24] the value of total dislocation density $\rho_{\rm T}$ for $\tau = 60$ MPa, $\rho_{\rm T} = 2.48 \times 10^{10}$ cm⁻², and the mean cell size $x_t = 1.19 \ \mu\text{m}$, from Fig. 3 on the Ungar *et al.* paper [24], by using Eq. (1) a value $u = 0.85 \ Gb^2$ it is obtained, and with Eq. (9), $u_{\rm T} = 0.87 \ Gb^2$. So the stored energy by dislocations is $\rho_{\rm T} u_{\rm T} = 0.65 \times 10^{-3} \ \text{J/mm}^3$, with a percentage difference with the previously determined value (4.69 J/mol) lower than 2%.

On the other hand, a supersaturation of vacancies can be created in a metal by energetic particle irradiation, quenching or mechanical deformation. At temperatures where the vacancy is mobile (generally $\geq 0.3 T_{\rm m}$), these vacancies can migrate and coalesce to form microscopic clusters [68]. According to a theoretical study about stability vacancy clusters in metals due to Zinkle et al. [68] the stacking fault tetrahedron, SFT, is the vacancy cluster energetically more stable than any other vacancy-cluster morphology (for a number of vacancies lower than 10^3 , here we have 79 vacancies in each SFT). By using this theoretical analysis it is easy to show that for the Rönnpagel et al. data [44] the energy stored $U_{\rm SFT}$ by SFT per unit volume is $U_{\rm SFT} = 4.5 \times 10^{-5} \text{ J/mm}^3$ which is 6.9% of the experimental stored energy by point defects. Then SFT are not capable alone to explain the main fraction of this energy and we need to search in other direction. Another possibility is that the "missing" stored energy arises from the stored energy due to vacancies created by plastic deformation (this stored energy depends on τ^3) and a fraction of which could remain inside the crystal under metastable experimental conditions. The creation rate of vacancies according to Saada (as cited by Kovács et al. [53]) is given by $\dot{n}_{r}^{+} = (0.3 \rightarrow 0.9)\sigma \dot{\epsilon}/(Gb^3)$. Then taking a mean value for the numerical constant equal to 0.6, and using experimental data from Fig. 6 on the Rönnpagel et al. paper [44] allows us to obtain the total vacancy density nv_p created from the beginning of plastic deformation until the shear stress reaches 60 MPa. $nv_p = 5.26 \times 10^{18} \text{ vac/cm}^3$. If we take into account that according to Friedel [69] the creation energy per vacancy is $Gb^3/5$, we have a maximum possible stored energy by vacancies equal to (6.82 ± 3.41) J/mol. This value is 14% higher than the determinedvalue for the stored energy by point defects. With the results of this section we are able to give, for the Rönnpagel *et al.* data, a possible explanation about the stored energy by dislocations, according to the soft and hard region model and also the stored energy in defects different from dislocations by using Friedel and Saada models.

The soft and hard region theory, which is a long range internal stress theory, has been able to explain:

- a. The basic mechanisms which lead to the formation of dislocation subgrains, in a qualitative way. Also the mechanisms which give rise to a dissipative subgrain wall structure during steady state. The analysis leads to a quantitative expression which relates the subgrain diameter with the applied stress for steady state power-law creep. For the first time the value of the well known phenomenological "constant", K, which appears in such relation is obtained as a function of basic parameters of plastic deformation, and it is established that the percentage difference between theoretical and experimental value for typical metals is 10% [11].
- b. The time evolution on dislocation density in a sigmoidal creep curve for Cu-16 at. % Al at 723 K, $\sigma = 39.2$ MPa before and after the subgrain formation begins (for times lower than 270 minutes). (See Sect. 3).
- c. In a full way, fundamental aspects of the experimental work on the stored energy in copper single crystals (See [24] and the first part of Sect. 4).

As far as we know, not one of these three problems already mentioned has been solved unambiguously by the mesh length theory or similar models. However, at this stage, it is clear for the authors of this paper that the controversy existing between the two big theories of work-hardening (soft and hard region theory and mesh length theory) requires a critical experimental test which unambiguously confirms the validity of only one of these theories.

Our proposal for such a critical experimental test is the following:

To make temperature measurements on Cu-16 at. % Al samples which are deforming in conditions where the dislocation structure evolves from a system composed of only isolated dislocations to one formed by isolated dislocations plus subgrain formation. According to the long range internal stress theory (soft and hard region theory) the process of subgrain formation is an irreversible dissipative process in which the mobile dislocation system evolves from a disordered (no subgrains) to a more organized structure (subgrain formation) under the action of an externally applied stress by means of a highly dissipative process (without any annihilation events of dislocations at the first stage of the subgrain formation process).

On the other hand, the mesh length theory is an equilibrium thermodynamical theory, and the beginning of the subgrain formation for such type of theories is not associated to an increment on the temperature of the sample.

According to the soft and hard region theory, the increase on the temperature of the sample must be localized in time as compared with the time required by the sample to exhibit the beginning of the subgrain formation process. The measurement of this increase on samples temperature can be done by using a calorimeter system similar to the one used by Mughrabi *et al.* for the case of copper single crystals [24]. The preparation of Cu-16 at. % Al samples and the observation of dislocations must be done following the procedure described by Hasegawa *et al.* [36]. Another possibility is to make observations on LiF

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where data about sigmoidal creep curves already exist [70]. For this case dislocations can be decorated by using different techniques [71-73].

5. CONCLUSIONS

In this work, a study about the subgrain formation process has been made by searching for its origin in terms of free energy. The analysis of the previous sections leads us to the following considerations:

1. The soft and hard region theory for the internal stress used together with some elements of linear elasticity allows to show the capability of the mobile dislocation system to evolve from a disordered to a more organized structure, (subgrain formation) under the action of an externally applied stress, by means of a highly dissipative process. This situation is in full agreement with the theory of self-organization in non-equilibrium irreversible systems. On the other hand, it is quite different to the case of a microscopically reversible and isolated system of dislocations which tends to states of maximum entropy, *i.e.*, of maximum disorder; with a minimum of free energy as it is the case of the mesh length theory.

2. For the case of sigmoidal transient creep in Cu-16 at. % Al at T = 723 K and $\sigma = 39.2$ MPa, our theory allows us to give a quantitative explanation of the time evolution of the mobile dislocation density before the subgrain formation starts. And also on the total dislocation density evolution after the subgrain formation starts but annihilation events have not yet appeared. When the subgrains begin to appear, thirty five percent of the total stored energy by mobile dislocations is dissipated by a sudden decrease of the mean energy of dislocations per unit length. This process occurs without any annihilation event of dislocations, which appear after the beginning of the subgrain formation.

3. Finally, in addition to the explanation of Rönnpagel *et al.* for the stored energy by dislocation arrangements in copper single crystals (by using a Soft and Hard Region Theory), we are able to explain the remaining fraction of the stored energy by considering the creation of vacancies during the deformation at room temperature. In this way, the total stored energy in copper single crystals deformed at room temperature has been explained by using the soft and hard region theory for the internal stress.

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