DEFORMATION BEHAVIOR OF MOLYBDENUM SINGLE CRYSTALS AS OBTAINED FROM QUASIRELAXATION *IN SITU* EXPERIMENTS IN A HIGH VOLTAGE TEM

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Abstract

A novel methodology for material characterization at the quasi-relaxation regime is presented. The dislocation dynamics on Mo single crystals was studied using experimental relaxation data for two stages of its development. The thermoactivated parameters of dislocation motion in single crystals are fundamental to identify the elementary mechanisms that control dislocation slipping under quasi-relaxation conditions. However, the thermo activated analysis to study the dislocation evolution, is not enough to absolutely identify the process itself. Hence, other methods are required. Thick specimens were tested by *in situ* HVTEM (High Voltage Transmission Electron Microscopy). As a starting point of non-elastic behavior in the metal, the yield point limit was considered, and modeling in quasirelaxation conditions was developed. Finally, the obtained data and analysis allowed us to reveal that the quasi-relaxation method is a powerful resource to obtain important information about the evolution of dislocations in metals.

Keywords: *Single crystal, In situ HVTEM, Quasi-relaxation method, Dislocations, Stress relaxation, Dislocation density.*

Resumen

Se presenta una novedosa metodología para caracterizar materiales en régimen de cuasi-relajación. Usando los datos experimentales de cuasi-relajación se estudia la dinámica de las dislocaciones en monocristales de Mo para dos etapas de este desarrollo. Los parámetros termo-activados del movimiento de dislocaciones en monocristales son fundamentales para identificar los mecanismos elementales que controlan su deslizamiento bajo condiciones de cuasi-relajación. Sin embargo, el análisis termo activado para el estudio de la evolución de las dislocaciones no es suficiente para identificar el proceso en si. Muestras gruesas fueron ensayadas *in situ* en el Microscopio Electrónico de Transmisión de Alto Voltaje (HVTEM). Se consideró el límite de fluencia como punto de partida para el estudio del inicio del comportamiento no elástico en el metal. Finalmente, la información obtenida permitió demostrar que el método de cuasi-relajación es un recurso muy poderoso para obtener información importante acerca de la evolución de las dislocaciones en los metales.

Palabras Claves: *Monocristales, In situ HVTEM, Método de cuasi relajación, Dislocaciones, Relajación de esfuerzo, Densidad de dislocaciones.*

1. INTRODUCTION

Since the 1930's there was knowledge of the very important role played by the dislocations produced during plastic deformation on the mechanical properties of metals. However, the studies for establishing the physical characteristics observed in the deformation curve had serious deficiencies. The mechanical characteristics, such as yield limit, deformation resistance, and the fracture limit, were correlated to the dislocational structure, which up to that time was studied by electron microscopy under static conditions [1]. *In situ* HVTEM (High Voltage Transmission Electron Microscopy) widened the possibilities in the study of the evolutionary processes inside the metal. In the present work the mechanical characteristics in micro-specimens and the structural rearrangement mechanisms are analyzed in dislocational systems under stress, characteristic of transition metals with BCC structure [2].

2. EXPERIMENTAL PART

The Mo single crystals were obtained by electron-beam plasma deposition in vacuum of at least 10^{-6} Torr. The impurity content of the single crystals was determined through mass spectrometry with a detection limit of between $10^{-6} \div 10^{-8}$% in mass, and corresponded to: C – 0.028; O – 0.02; N – 0.006; H – 0.002; Mg – 0.001; Si – 0.003; Ni – 0.001; Al – 0.001; Fe – 0.003; W – 0.1, % in weight. According to Han *et al*. [3] the impurity content measured implies a monophasic system; a small percentage of inclusions of Mo_2C, oxides of Mg, Ca, Si, Al and nitrides is also present [4].

Molybdenum single crystal micro-specimens with $[100]\,(0\bar{1}1)$ and $[100]\,(001)$ orientations, were tension tested inside the HVTEM with ultimate tension strength equivalent to the dislocation segment tension between 0.1–4.8 kg/mm^2, *i.e.*, close to σ_μ, the micro-yield limit, as determined from the quasi-relaxation tests [5]. Hence, the micro-yield limit is the stress level that induces dislocation movement.

The structural symmetry was analyzed by X-ray analysis and HVTEM. The Laue diagram analysis of the reflecting surfaces type {112} and {310} allowed the sub-grain evaluation in the radial and azymuthal (Bragg and anti-Bragg), directions respectively.

Planar single-crystalline specimens were tested with an area of (0.7 to 1) x 3 mm^2, and 20 mm length. The specimens were tested under quasi-relaxation regime in an INSTRON, series *4500* machine, with a registry precision of 0.5%, and a deformation velocity up to quasi-relaxation regime of 4 x 10^{-4} sec^{-1}. The results of the INSTRON were plotted in "log (σ) vs. t" coordinates.

3. RESULTS AND DISCUSSION

The relationship between the mechanical characteristics and the final structure parameters in a metal is well known, in particular the functional correlation between deformation vs. dislocation density ρ, which can be expressed by:

$$\tau = \alpha\, G\, b\, \rho^{0.5} \qquad (1)$$

where ρ is the dislocation density, α is the dislocation multiplicative coefficient, dependent on a number of physical conditions, G is the metal's shear modulus, and b is the Burger's vector.

In particular equation (1) was obtained by several investigators, to determine the yield point strain in crystalline materials based on different models derived from the analysis of the sliding dislocations interactions with barriers of varying nature, that come on the way of free movement of the dislocations during the transition from elastic to plastic deformation. The physical meaning of the other parameters in the equation, depends strongly on the particular mechanisms considered as a model to explain the interaction of the dislocations with the barriers during the plastic deformation process; in some cases ρ refers to dislocation density at the walls of the crystalline structure; in a second it refers to the density of a dislocation forest and in another case as generalized slipping dislocation density.

In Table 1, the most important parameters for this interactions and their physical meaning are shown; their understanding allows a better grasp of this problem.

In equation (1), or it's analogue:

$$\tau = \tau_o + \alpha\, G\, b\, \rho^{0.5} \qquad (2)$$

we can introduce the experimental results obtained from the study of τ vs. $\rho^{0.5}$ for a series of materials. Table 2 shows some of the theoretical and experimental results related to stress and dislocation density analysis, and the main difference observed is only on the value of the α coefficient [6-8].

Nevertheless the analysis of $\tau = f(\rho^{0.5})$, from mechanical tests followed by HVTEM reveal a significant contradiction in the physical treatment. In particular extrapolation of this function to a square root type of initial dislocation density, in many occasions leads to negative values of τ_o [8,9], that has no physical meaning whatsoever. For instance, when Nb single crystals are deformed, an increase in the resistance to deformation has been observed; at the same time, for τ_o, determined from the function $\tau = f(\rho^{0.5})$, these values are reduced [9].

In a similar way, this lack of correlation was detected for cyclic tests results [10], where the yield stress indirectly determined from the stress-strain deformation diagram yielded smaller values in comparison with static tests, where extrapolation of the $\tau = f(\rho^{0.5})$ leads to opposing conclusions. According to Nabarro *et al*. [11] equation (1) can be obtained from magnitude analysis.

Table 1. Physical meaning of the parameters in equation (1), and model of dislocation interaction with barriers.

α in equation (1)	Meaning of ρ	Interaction mechanism of dislocation	Author of proposed model
$\dfrac{1}{2\pi}$	Dislocation density in the limits of crystalline lattice	The sliding dislocations surpass the resistance generated by the dislocations that generate periodic networks of dislocations	Taylor [1]
$\dfrac{1}{2\pi}$	Density of sliding dislocations	The sliding dislocations surpass the effect of containment of the barriers generated on them as a result of the intersection with the dislocation nucleus.	Hirsh, Mott [2]
$\dfrac{\sqrt{n}}{2\pi}$ for $n = 25$, $\alpha = 0.795$	Concentration and dislocation density, n is the number of dislocations in planar concentration	The sliding dislocations overcome the long range stresses arising from the planar concentration of dislocations	Zeeger [3,4]
$\dfrac{1}{\pi}$	Density of the dislocation forest	Deflection of the sliding dislocations in the dislocation forest.	Kulman - Wilsdorf [5]
$\dfrac{1}{2\pi}$	Density of the dislocation forest	Resistance to the sliding dislocations from the intersection of the dislocation forest forming steps	Bazinsky [1]
$\dfrac{1}{2\pi}$	Total dislocation density	Overcoming the mutual attractive forces of the dislocations for a relative displacement	Gale B [6]

Table 2. *α* from theoretical and experimental results [7,8]

Metals	T (K)	Values of α in equation (2)	
		Single crystals	*Policrystals*
Cu	4.2, 293	0, 38; 0.38, 0.21	0.5
Ag	293	-	0.58
Fe	138-198	0.5-0.6	0.7-0.8
	300	0.30	0.70, 0.50, 0.38
	373-473	-	0.80
Mo	77	1.42	-
	195-200	0.84, 0.35	0.36
	300-373	0.49, 0.25	-
Nb	138-198	0.375	-
	293	-	0.42, 0.47,
	300-523	0.125	0.425, 0.440,
V	230	-	0.36
	293	-	0.43
	300	-	0.27
	673	-	0.21
Ta	373	0.3	-

The objective of this work was oriented to demonstrate that the $\tau = f(\rho^{0.5})$, functionality can be obtained not only from formal considerations of the magnitude theory, neither from particular models of the interrelation of the sliding dislocations with barriers of different nature, but based on general physical laws on energy conservation, presenting results of a series of analysis obtained from quasi-relaxation tests performed *in situ* on Mo single crystals inside the HVTEM.

Equation (1) can be obtained from simple energy considerations and it describes the transformation of an initial dislocation structure to one of a specimen under load, that is reversible related to the original structure when the load is applied rapidly, but it can evolve slowly in an irreversible manner under quasi-relaxation conditions.

Studies of thin sections of micro specimens in tension of single crystals of Mo, W and other single crystals with an analogous crystalline geometry to the ones presented in this work (but here tested in the quasi-relaxation regimen *"in situ"* in the HVTEM JEOL *1000*, starting from thicker specimens,) demonstrated that the initial physical explanation of the elastic energy accumulation process during load application requires a correction for thick ("real") specimens.

The micro-specimen deformation measured by the emergence of dislocations at the specimen's surface demonstrated that only part of the plastic deformation is related to the reversible movement of the dislocations for an applied stress below the yield limit. The other one is, however related to the fact that when a load is applied and the specimen deformed, the interatomic distance expands or increases. It is not difficult to demonstrate that the presence of a dislocational elastic component in the deformation, reflects itself on the magnitude of the elastic relaxation modulus of the specimen more intensely the higher the relationship between the dislocational and atomic component of the deformation, and it can be expressed as:

$$E_r = \frac{E}{1 + \dfrac{\varepsilon_d}{\varepsilon_e}} \qquad (3)$$

where E is the Young's modulus of the metal, E_r is the Relaxed Young's Modulus of the metal, and ε_d is the dislocation deformation; ε_e atomic (lattice)

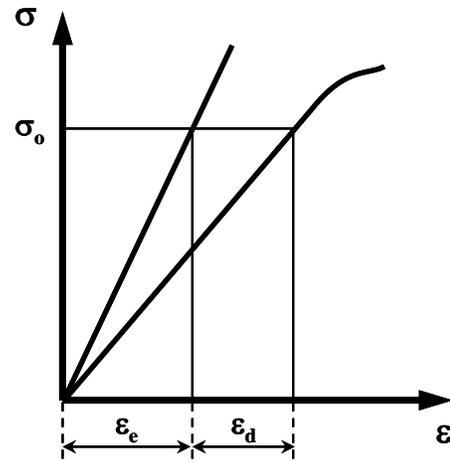deformation; this is shown schematically in Figure 1.

Figure 1. Schematic representation of equation (3): ε_e is the contribution to elastic deformation by the induced geometrical lattice distortion. ε_d is the contribution to the deformation in the elastic range by dislocation displacement during the load in the elastic range.

From Figure 1, it can be inferred that:

$$\varepsilon_e - \sigma = E\,\varepsilon_e = E_r\,(\varepsilon_r + \varepsilon_d) \qquad (4)$$

and,

$$E = E_r\,(\varepsilon_r + \varepsilon_d)\,/\,\varepsilon_e, \qquad (5)$$

after division by ε_e.

When the crystal is deformed elastically, the dislocations are elastically deformed too, meaning they stretch out, increasing their Burgers vector. As a consequence the elastic modulus changes, and this fact has to be taken into account when we determine the relaxation modulus. Notwithstanding, this is not large as compared to the contribution from, or attributable to, the whole load loop of the testing machine.

Considering that the scheme proposed here is incompatible with the traditional method, and ultimately it is based on experimental methods that allow the direct determination of some physical parameters, that characterize the dislocational structure, we modernized the methods according to the fundamental proposals of our scheme.

The equation that describes the dynamical

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dislocation kinetics under quasi-relaxation conditions is very complex and can be expressed by [12-14]:

$$\rho(t) = \frac{2}{G\,b^2}\left(U_e - \int_0^t \sigma(t)\,d\varepsilon(t)\right) \qquad (6)$$

where U_e is the initial elastic energy in the loaded specimen up to a maximum stress σ_o.

The analysis of the quasi-relaxation experiments on Mo were the initial point for the study of the evolution of the elementary thermally activated processes of the dislocational structures.

We only evaluated the influence of the proposed correction for the case of an initial dislocation density in the specimen up to the moment when the machine is stopped, (which corresponds to the point when the stress micro-stage inside the microscope is stopped, for instance at 0.6 the yield stress) which considers the two components of the elastic deformation.

The method developed here by us to analyze the quasirelaxation process is characterized by its richness in data; the whole series of experiments were done using a single specimen that was tested at different initial stress levels. That allowed us to obtain enormous quantities of information about the dynamics of dislocations in the tested specimens, about the length of their displacement, information about the trajectory of this displacements, information about the elementary thermo activated processes, of the evolution of the dislocational system, about some specific mechanical properties related to the inelastic behavior of the metal (micro yield limit and proportionality). Some of the possibilities that these methods allow are applied here for Molybdenum.

If we assume that all the energy concentrated inside the specimen in the elastic range, is accumulated in terms of the energy of the dislocations` elastic fields then:

$$\frac{\sigma^2}{2\,E_r} = \frac{G\,b^2}{2}\rho_d \qquad (7)$$

where ρ_d is the dynamic dislocation density. An expression can be worked out that allows the determination of the initial dislocation density in the specimen before application of the load on the

simple. When the specimen is loaded inside the elastic range, then it is assumed that the specimen already had dislocations. If we put $\alpha = (E_r / G)^{0.5}$, then equation (7) can be expressed as equation (1), considering $\tau = \sigma/2$.

In Table 3, the values for a series of metals of the α coefficient are shown determined for specimens with 5 mm diameter and 50 mm length, for a loop of $S = 2.6 \times 10^{-4}$ cm/kg, according to:

$$E_r = \frac{l_o}{F_o \sum_{i=1}^{n} C_i} \qquad (8)$$

where l_o is the initial length of the specimen's work zone, F_o is initial area of the work zone, and C_i is the elastic behavior of the force elements of the test machine (grips, grip lengthening, the column, etc).

In effect, the accumulated free energy of the dislocations is equal to the difference between the elastic energies of the whole loading system and the elastic energy of the specimen, released during discharge, hence:

$$\Delta g = \frac{\sigma_o^2}{2\,E_r} - \frac{\sigma_o^2}{2\,E} \qquad (9)$$

Δg is the Gibbs free energy of the specimen under tension.

It is important to stress that the coincidence between the data in Tables 1 and 3, is much better than the data in Tables 1 and 2. A more in depth analysis of the problem can be found in Ref. 13 (see Ivanova and Ermishkin, p. 80).

3.1 Energy Considerations of the Problem

For this case the increase in the free energy of the specimen as a result the accumulation of a high density of dynamic dislocations can be expressed by:

$$\Delta g_o = \frac{\sigma_o^2}{2\,E_p} - \frac{\sigma_o^2}{2\,E} = \frac{\sigma_o^2}{2}\left(\frac{E - E_p}{E_p\,E}\right) \qquad (10)$$

$$g_o = \frac{\sigma_o^2}{2\,E_p}\rho_o = -\frac{\sigma_o^2}{2}\left(\frac{E - E_p}{E_p\,E}\right) \qquad (11)$$

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Table 3. *α* determined experimentally for several metals.

Metal	α in equation (1)	Metal	α in equation (1)
W	0.116	Cu	0.202
Mo	0.130	Al	0.254
Nb	0.188	Ti	0.221
Fe	0.146	Ni	0.156

Table 4. Influence of the elastic deformation of the dislocations on the dynamical dislocation density and the *α* coefficient.

σ_o (MPa)	E_p (MPa)	α		$\rho/10\,(cm^{-2})$	
		$\alpha_1 = \sqrt{\dfrac{E_p}{G}}$	$\alpha_2 = \sqrt{\dfrac{E_p\,E}{(E-E_p)\,G}}$	ρ_{o_1}	ρ_{o_2}
225	2492	0.143	0.143	2.25	2.25
180	2556	0.145	0.145	1.40	1.40
135	2794	0.151	0.152	7.26	7.16
90	3270	0.164	0.165	2.73	2.70
45	4444	0.191	0.192	5.64	4.99

Equation (11) implies:

$$\sigma_o = \alpha\,G\,b\,\rho_o^{\,0.5} \qquad (12)$$

$$\alpha = \sqrt{\frac{E_p\,E}{(E-E_p)\,G}} \qquad (13)$$

Equation (12), without consideration of the elastic deformation of the dislocations, can be written as:

$$\alpha = \sqrt{\frac{E_p}{G}} \qquad (14)$$

Table 4 presents data that shows how the elastic deformation of dislocations influences the density of the dynamic dislocations for well established initial boundary conditions and values of the stress σ_o in a Mo single crystal [110]<100> at T = 293 K.

From Table 4 we realize that to consider the influence of the elastic deformation of the dislocations on the magnitude of the *α* coefficient and the density of the dynamic dislocations for pre-established conditions of the initial stress, does not influence strongly the value and can be discarded for all practical purposes. For that reason, the initial arguments presented in [12,15], are undoubtedly attained from the qualitative and quantitative points of view, as much as regards the evolutional image of the dislocational structure during quasirelaxation as well as their evaluation characteristics respectively.

Figure 2 shows quasirelaxation curves obtained for different initial stress σ_o at T = 293 K. In these curves two stages are clearly defined: The first stage lasts up to 50 seconds and the second stage manifests itself during 50 to 700 seconds of the start of the test.
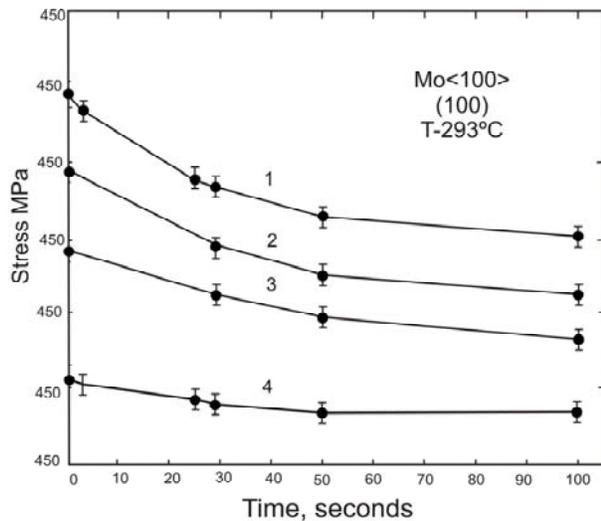
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Figure 2. Quasirelaxation curves for Mo single-crystal specimens. The values correspond to (1) 396 MPa, (2) 296 MPa, (3) 292 MPa, and (4) 208 MPa.

In previous work [13,15] we have demonstrated that the whole quasi-relaxation curve can be analytically described by the superposition of two functions a hyperbolic and an exponential function of the kind:

$$\sigma(t) = \frac{\sigma_0 - \sigma_1}{1 + K(\sigma_0 - \sigma_1)t} + \sigma_1 \cdot e^{-\gamma t} \qquad (15)$$

where σ_o, σ_1, K, and γ are approximation parameters.

Beyond 30-120 seconds of the test (within the margin of error) the experimental curve corresponds to an exponential behavior. The statistical verification of the hypothesis on the possibility of representing analytically the relaxation experimental curves, given in equation (12) and the exponent using the Peerson criteria, allow us to sustain this method.

The single crystal substructure with growth axis <100> contains first order sub-grains with illed defined columns de 1 a 2 mm in length and $0.1-0.5$ mm in transversal direction, and also second order equiaxial sub-grains with sizes between $0.05-0.5$ mm. The miss orientation between the subgrains of first order is bellow tenths of a degree, and between subgrains of the second order less than 0.15 of a degree (Figure 3).

The observed structure shows different dislocation segments, stretched out in the <111> direction, dislocation concentration and small angle dislocation edges. The average dislocation density

ρ, oscillates between 10^6-10^7 cm^{-2}.

Mo, W, Fe, FeSi, Va and Zn single crystal micro-specimens and alloys of W+Re and Ta *in-situ* tested in the HVETM column, have demonstrated that the edge dislocations start moving at very low stress values, several times lower than the proportionality limit determined by the tests done in macroscopic specimens in universal testing machines. This circumstance coincides with the macroscopic experimental data about the study of dislocation mobility at room temperature using the method of selective corrosion under load, according to the three point bending method. Dislocation movement was observed at a value of $\sigma = 0.40$ kg/mm^2 for a Mo [17], and $\sigma = 0.56$ kg/mm^2 for W [18].

In the present experiments, in [100] $(0\bar{1}1)$ and [100] (001), single crystal Mo shows that the ultimate tensile stress, determined approximately from the dislocation segment tension [6], has a value between 0.1–4.8 kg/mm^2, *i.e.*, very close to the micro-yield limit values σ_μ, determined from quasi-relaxation experiments and hysteresis mechanisms.
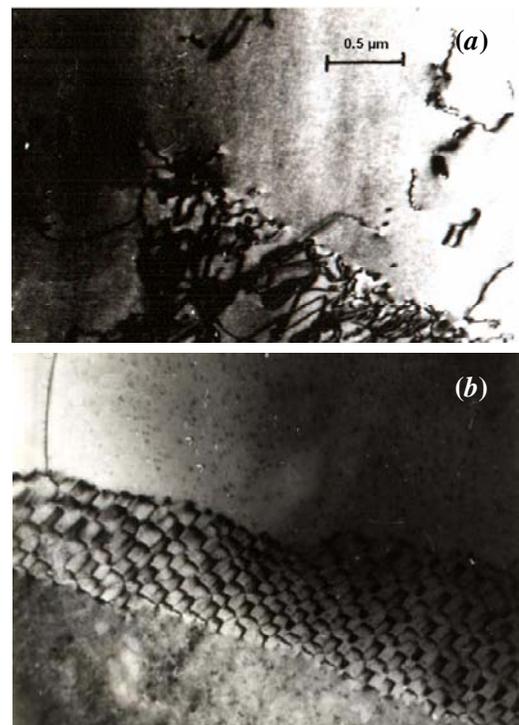

Figure 3. As-grown dislocation structure of a Mo single crystal: (*a*) Dislocation concentration, and (*b*) Sub-boundaries.

The equation that describes the deformation process of the specimens under quasi-relaxation conditions, was developed by us considering the following conditions: the length of the load loop of the testing machine is kept constant after turning off the system at a relaxation instant $t = 0$, and in another moment t; in the most general case, the specimen begins to relax after a previous plastic deformation ε_p was performed. Hence, starting from the boundary condition that during the test the longitude of the mechanical force loop of the machine is kept constant, it is possible to obtain an expression for the deformation in the structure with the quasi-relaxation behavior that can be represented by:

$$\varepsilon = \left\{ \sigma_0 - \sigma(t) \left(1 + \frac{2\,\varepsilon_n\,C_0}{\sum\limits_{i=1}^{n} C_i} \right) \right\} \frac{\sum\limits_{i=1}^{n} C_i\,F_o}{l_o} \quad (16)$$

where σ_o is the initial stress level, ε_o is the initial deformation of specimen, $\sigma(t)$ is the stress applied on specimen at time t, and $\sum C_i$ is the elastic coefficient of the mechanical loop considering the specimen elastics, and C_o is the rigidity of the specimen.

Equation (16) is obtained from the comparison of the length of the loop of load of the test machine immediately after applying the stress on the specimen up to a value σ_o, at $t = 0$, and then at a time t; it is assumed that at t is plastically deformed with a deformation ε_o.

At $t = 0$, the length of the loop of load of the test machine (L) is equal to the sum of all the elongations of the machine elements that transmitted the load on the specimen; i.e. to the length of both grips, including the specimen, which are deformed both elastically as well as plastically ε_o. If we designate the elastics of the whole machine as S_i, then:

$$L(o) = \sum_{i=1}^{n-1} l_i + P_o \sum_{i=1}^{n-1} C_i + \frac{P_o\,l_o}{F_o\,E} + \varepsilon_o l_0 \quad (17)$$

and after a time t, of stopping the machine, the same length can be expressed as:

$$L(t) = \sum_{i=1}^{n-1} l_i + P(t) \sum_{i=1}^{n-1} l_i\,C_i + \frac{P(t)\,l_t}{F_t\,E} + \varepsilon_o l_0 + \Delta l_r \quad (18)$$

being Δl_r the increase in the specimen length during the process of stress relaxation. Because the machine's loop length cannot vary, and

$$L(o) = L(t) \quad (19)$$

Equating the right hand side of equations (17) and (18), and upon reduction we obtain:

$$(P_o - P(t)) \sum_{i=1}^{n-1} C_i = \frac{P(t)\,l_t}{F_t\,E} - \frac{P_o\,l_o}{F_o\,E} - \Delta l_r \quad (20)$$

Considering that the specimen's volume is kept constant during the plastic deformation, then $l_o\,F_o = l_t\,F_t$, and hence:

$$F_t = l_o\,F_o / l_t \quad (21)$$

During load, the specimen's elastic behavior, after an initial plastic deformation, can be obtained by mixing equations (8) and (21), which leads to equation (22):

$$\frac{l_t}{F_t\,E} = \frac{l_t^{\,2}}{l_o\,F_o\,E} = \frac{(l_o + \Delta l_p)^2}{l_o\,F_o\,E} \approx \frac{l_o^{\,2}(1 + 2\,\varepsilon_o)}{l_o\,F_o\,E} = \frac{l_o(1 + 2\,\varepsilon_o)}{F_o\,E} \quad (22)$$

After putting equation (22) in (20), and calling $\varepsilon = \Delta l_r / l_o$, followed by simple transformations the result acquires the following functionality:

$$\varepsilon = P_o \sum_{i=1}^{n} C_i - P(t)\left(C_i + 2\,C_o\,\varepsilon_o\right) \frac{F_o}{F_o\,l_o} = \left\{ \sigma_o - \sigma(t)\left(1 + \frac{2\,C_o\,\varepsilon_o}{\sum\limits_{i=1}^{n} C_i}\right) \right\} \times \frac{F_o \sum\limits_{i=1}^{n} C_i}{l_o} \quad (23)$$

i.e. equation (23), is the expected result.

In Ref. [12] it was demonstrated how it is possible to analyze the dislocations dynamics and realize a thermally activated analysis of the deformation under quasi-relaxation conditions using equations (1) and (2). *In situ* three point flexural bending tests, showed that the dislocations sliding start at very low stress values, several times lower than the proportionality limit as determined from the macroscopic tests, as mentioned above for Mo [15]. The stress that produces the break-up of the dislocation segments depends on the chemical purity of the metal and the dislocation's segments length. Considering that at the moment of edge dislocations breakup, the initial dislocation density ρ can increase by a factor not higher than $\pi/2$, and from the equation (1), then for Mo with starting ρ of 10^5–10^6 cm^{-2}, the micro-yield limit fluctuates between 0.05 to 0.15 kg/mm^2, which is very similar to the stress necessary to put into motion the screw dislocations. As a consequence, even in the plastic deformation regime, for macroscopic specimens, $\sigma_{pr} = 14 - 18$ kg/mm^2, an intense dislocation generation is produced by the accumulation of the tails of the non-screw mobile dislocation segments edge. When the deformation starts, the screw dislocation multiplication is the most efficient mechanism to increase ρ in micro specimens, as can be seen in Figure 4. To this mechanism corresponds up to 80% of the increase in mobile dislocation density.
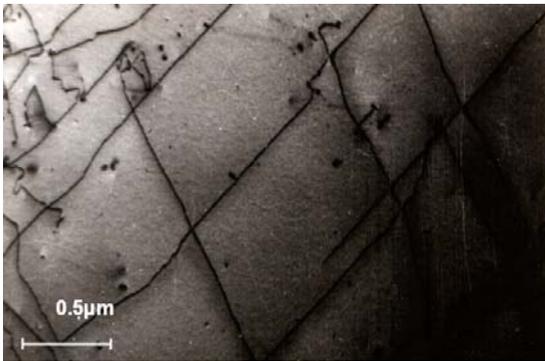

Figure 4. Dislocation structure in a Mo single crystal weakly deformed.

The remaining 20% corresponds to regenerative mechanisms of new dislocations; this mechanism is the Frank-Reed, with an anchoring spot, as shown in the schematic Figure 5, also to dislocation sources that act on stress concentrators at the specimen edges or in micro-cracks, as shown in the HVTEM micrograph (Figure 6).

At the start of the deformation process, when the screw dislocations increase dynamically, which were initially generated by the sliding of the edge segments of type J and V, the increase in the moving dislocation density is also influenced, and when a micro-crack is present, the regenerative type sources prevail.

The results of these observations coincide with those of Japanese researchers which have confirmed many of the characteristics about the dynamic behavior of the individual dislocations and their inter-relationships, which were theoretically predicted [16,17]. When the load is increased, the number of edge dislocation segments increases which were involved during the deformation process and hence in a very intense way develops the increase in the screw dislocations density.

In the early stages deformation of the specimens, the sliding of dislocations in <111> {110}, was observed in which the planes of type {110} were show-cased.
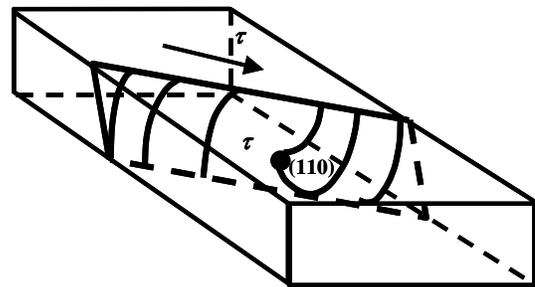

Figure 5. Schematics of the Frank-Reed source with an anchoring point.
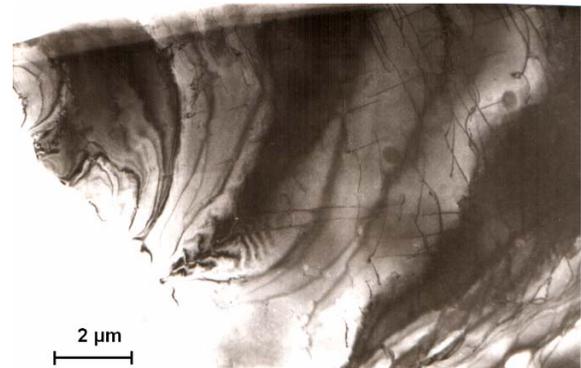

Figure 6. In situ observation of dislocation generation at the front of crack of concentrated stress in a Mo single crystal.

The dominant role of the non-screw (edge) dislocations, on the plastic deformation of micro specimens in the early stages of deformation, allows an association to the instant at which the micro yield limit appears. At that instant the non-screw dislocations break up and the massive sliding starts of the screw dislocations, originating the emergence of the yield tooth as a step of the micro yield [18,19].

The sudden increase in ρ of the dynamic dislocations at the stage, which from the view point of the macroscopic deformation laws define elasticity, allows an analysis as a stage of accelerated process of hardening. Problems related to the hardening coefficient at this stage and the relationship between stress and strain were analyzed before in [20] and by Yermishkin and Plastinin [23] for Fe. It is interesting to point out that Imura *et al.* [2] came to the same conclusions on single crystals of Fe. In the above references $\tau / \tau_s = f(\rho)$, describes the sliding dynamic dislocation density, which increases in well defined temperature ranges that for single crystalline Fe varies between $T = 153\ K$ and $293\ K$ at the micro yield stage up to reaching the yield limit. It can be noticed that the dislocation multiplication in single crystals of Fe occurs at least, at a stress of $\tau / \tau_s \geq 0.25$.

As a support of the developments presented here, in Figure 7, $\tau = f(\rho_{dyn}^{0.5})$ is plotted for Fe specimens, using data obtained from tests done by the quasi-relaxation method, in which the experimental data from [2] has been used. The agreement between the data obtained from quasi-relaxation and that obtained by *in situ* HVTEM is remarkable so much qualitatively as quantitatively. Hence, we can point out without hesitation that the results shown in this paper support strongly the idea that the active dislocation multiplication is characteristic, not only of the macroscopic plastic deformation, but also of the micro plasticity stage [21]. If in the micro plasticity stage the deformation velocity is expressed $\varepsilon = b\rho v$, and is given by an enhanced sliding velocity of the non-screw dislocations of relative low density ρ, then when the macroscopic limit of yield is reached, this will yield a slow sliding of the screw dislocations with a high density ρ.

As a consequence of the above, the yield limit corresponds physically to the stress at which the

highest importance for the plastic deformation appears through the sliding of screw dislocations.

The emergence of the yield tooth in the stress-strain diagram is related to the fact that in absence of mobile dislocations, the disanchoring of sources that generate them requires considerable higher stresses than for the dynamic multiplication during the process of an ulterior deformation.
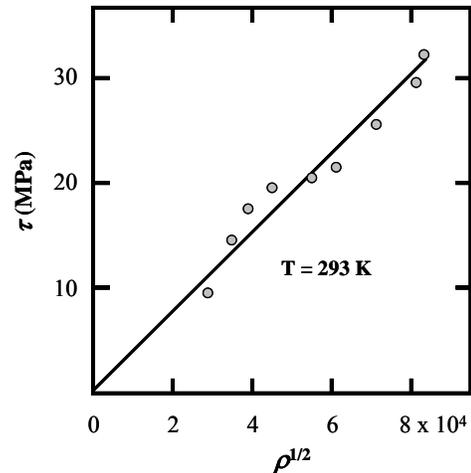

Figure 7. $\tau = f(\rho_{dyn}^{0.5})$ for single crystals of Fe, as measured from the quasi-relaxation data. Experimental points from Ref [2], at 293 K at the micro yield stage.

4. CONCLUSIONS

As results of HVTEM *in situ* experiment*s* it has been established that:

- During load above the micro yield-point, elastic deformation happens through the dynamic dislocations which appear as a result of reproduction of the original dislocations contained in the simple, when sliding is activated of the edge dislocation segments, dragging along the tails of the screw dislocations.

- The dynamic dislocations present in crystalline materials under load conditions, reversibly are related to the dislocation structure that stays in the specimen after unloading;

- Equation (1) relates the applied stress with the dynamic dislocations density under loading conditions, so much in the elastic field, as well as during the plastic deformation of the

specimen.

- The concepts established in this work avoid the contradictions between the theoretical analysis of the function $\tau = f\,(\rho^{0.5})$ and the analogous data obtained from the experiments;

- The results from the studies on the dynamics of the dislocations based on the relations obtained from experiments under quasirelaxation conditions, were corroborated experimentally *in situ* in the HVTEM using Mo single crystals with identical chemical composition and crystallography.

- The *in situ* tests inside the HVTEM allow establishing an excellent correlation of the evolution of the structure of dislocations at two levels of resolution: In the atomic range of the order of 0.2-0.5 μm, and at the macro range, *i.e.*, several microns.

5. ACKNOWLEDGMENTS

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