NUCLEATION OF MICRO-CRACKS IN FINE GRAINED TUNGSTEN AT THE AMBIENT

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Abstract
Crack nucleation was studied on primary recrystallized AKS doped tungsten at the ambient upon bending. Bending often generated non-propagating micro-cracks along short grain boundaries ending at a common triple line. However, the frequency of micro-cracks was not governed merely by geometrical factors such as the extent of grain boundary segments or their orientation with respect to the wire axis. This finding is in accordance with the expectation that crack-nucleation has to be sensitive also on the crystallographic parameters of the grain boundaries and on compatibility stresses, the magnitude of which depends also on the relative crystallographic positions of adjacent grains. An analysis of the micro-crack nucleation of as-drawn and stress relieved doped tungsten wires has led to the conclusion: a thin dislocation coverage on a grain boundary favours the nucleation of micro-cracks in various deformation modes. The reason is: grain boundary splitting is energetically facilitated, because the fresh surface created by splitting attracts dislocations, which are annihilated upon a short-path glide to the free surface.
Keywords: doped tungsten, compatibility stresses, working damage, micro-cracks

INTRODUCTION

Heavily drawn tungsten is ductile in the as-drawn condition in the following sense. It can be coiled onto a mandrel, the diameter of which is only twice as large as its diameter. Coiling does not lead to wire-break in the standard production, but macro-cracks are often formed during this operation, and they have an adverse effect on the performance. Stress relieved and primary recrystallized wires have also a certain ductility in bending, but they are also prone to crack formation.

Before the analysis of micro-crack formation, let us have a look at the fracture-mechanism maps [1] visualising the parameters at which the common fracture modes dominate. Although the published maps on BCC metals concern merely the fracture of equiaxial recrystallized structures in a tensile test, recent studies on recrystallized tungsten [3-6] determined also the strain rate effect in a broad range (10^{-3} s^{-1} and 10^{3} s^{-1}) and extended the data base to compression test [3], torsion [4] and bending [5], as well as to swaged rods with moderately elongated microstructures [4-5]. Unfortunately, it is not a trivial matter to fit the fracture modes of doped tungsten into the frame work of common fracture maps. It is usual to seek the reason of this difficulty in the ultra-fine microstructure that develops upon the commercial thermo-mechanical processing of incandescent filaments. The essential points are as follows. (i) True strains in excess of a 99% reduction

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in area are involved without intermediate stress relief [6-8]. (ii) The size of the as-drawn microstructure is ultra-fine (300 nm > d). (iii) The grain structure is fine also in the primary recrystallized condition (the transversal mean linear intercept is about 1 μm), although the grains are very elongated [9,10], since rows of ultra-fine sized potassium bubbles are formed in doped tungsten upon processing, which stabilise this fibrous grain structure.

What are the peculiarities of fracture in heavily drawn tungsten? It has long been known that this material shows a reduction in area from 40 to 60% upon necking in a tensile test at the ambient in the as-drawn condition [7,10]. This marked "ductility" is contrasted by the fact that the uniform plastic strain before necking ranges merely from 1 to 4% [7]. Thus, one should classify this famous “working induced ductility” as limited plasticity being typical for the domains of cleavage 3, or brittle intergranular fracture 3 of the fracture-mechanism maps. However, this classification should be preferred withstanding the marked necking, because the start of necking is simultaneous with the nucleation and limited propagation of longitudinal intergranular cracks [7,10-13]. (The lengths of the first cracks are less than one tenth of the wire radius [13].) Since these cracks are referred to as splits in the industry, we will often use this term later on, as well.

What happens, when the “working induced ductility” is lost upon annealing? The extent of uniform plastic deformation remains in the range of limited plasticity ($0.01 < E_u < 0.04$), even if heavily drawn samples are annealed between 300 and 1800°C [7]. Consequently, there is no loss of ductility upon annealing in terms of the common fracture mechanism maps. A ductility loss evolves merely in terms of the quality control of lamp grade tungsten that considers the reduction in area upon necking as the basic measure of ductility. The scenario of necking is as follows. This measure remains constant upon anneals below 800°C, decreases rapidly with increasing annealing temperature above 800°C, until it becomes virtually zero at 1300°C. The essential change in this scenario is: the extent of deformation with splitting becomes very short upon anneals performed in the said temperature region [7,8,10-13]. In order to settle the contradiction in terminology, one may prefer to consider the extensive necking in tensile test as a special kind of formability, in which splitting allows a rupture-less plastic deformation of marked extent, because splitting suppresses cleavage. When one is willing to adopt this point of view, then the absence of the extensive necking in stress relieved microstructures may be ascribed simply to the fact that a certain type of working damage can not be generated in a softer matrix.

This work was stimulated by the fact that tungsten and molybdenum are subject to extensive splitting not only in tensile tests but also in various modes of working like wire drawing, rolling or coiling. Therefore, splitting is a question unbroken as to technological interest [12,18-21]. Although the coilability at the ambient is in agreement with the finding that the ductile-brittle transition temperature of heavily drawn tungsten is below -10°C in bending [8], it is well established that coilability requires a more dedicated test, since as drawn tungsten often splits during coiling [12]. As splitting typically does not yield to separation of the work-piece into distinct parts [7-8,12], we should consider also wire splitting upon coiling as a peculiar form of working damage. Also extensive split-type working damage evolves before the failure of as- drawn tungsten, when it is tested in free-end torsion at the ambient [14-17]. Let us stress that the torsional working damage is connected with large failure strains. For example, the true shear strain at the surface of fine wires ranges usually from 0.8 to 2 at failure in free-end torsion at the ambient.

It is also well known that the split propensity depends on the details of the thermo-mechanical processing route in various modes of deformation [7-8,12,18], which prompted extended studies on its microstructural background, both in as-drawn and annealed heavily drawn tungsten. Also dedicated tests were devised for this purpose (bend-and-stretch test [22], knife-edge compression testing [23,24]). It turned out that the dispersed potassium phase plays only a minor role in splitting [22-24], while the role of the grain structure can be characterized as follows [23-25].

1. The correlation between the transversal mean linear intercept and split load is less pronounced in a knife-edge test. Thus, the longitudinal boundaries should play only a secondary role in crack nucleation, although the major part of a crack propagates along them.
2. The split load in the knife-edge test seems to be governed by the frequency of transversal boundaries.

What are the essential conclusions of these studies? It has long been known [26] that the mean transversal intercept of grain boundaries is an important parameter of the limited plasticity in any elongated microstructure as far as hardness is concerned. However, only recent indirect evidence supported the view that the cleavage nucleation should be governed by the transversal grain boundaries in a knife edge test. This interpretation is in accordance with the general belief that the well-known transition in a tensile test from transgranular fracture to the cleavage dominated fracture upon stress relief and primary recrystallization should be ascribed to transversal grain boundaries that are formed upon these treatments [11,25]. Unfortunately, no direct evidence has been published on micro-cracks that were formed on the transversal grain boundary segments in any of the common deformation modes, with the exception of a recent study in free-end torsion [17].

The present work aimed to reveal the micro-cracks formed upon the bending of primary recrystallized doped tungsten at the ambient, and intended to rationalise the different split propensity in as-drawn and primary recrystallized doped tungsten in various deformation modes.

EXPERIMENTAL

The samples in the bending test were 50 mm long pieces of commercial lamp grade tungsten having a diameter of 173 μm. They were bent with a half turn onto a cylindrical tungsten mandrel having a diameter of 1.5 mm. The true strain amplitude of the applied bending was 0.17. It was determined from the radius of curvature formed directly at the mandrel after load release.

The wire surface has been carefully cleaned through a two-step heat treatment. The first treatment was performed at 1200°C for 10 minutes in a high vacuum chamber having an adjusted O_2 pressure of 10^{-4} mbar, while the second treatment was carried out at 1800°C for 1 hour in the same ultra-high vacuum chamber having a residual oxygen partial pressure less than 10^{-11} mbar. The first treatment cleaned the free surfaces from carbon and carbon compounds and also removed the segregated carbon from the grain boundaries, while the second one removed segregated oxygen from the grain boundaries via oxygen diffusion and evaporation of WO_X gas molecules from the free surface.

The proof stress at 1% conventional plastic strain in the tensile test at the ambient was 1.6 GPa. The extent of necking was low, the fracture surface was stair-case like and consisted of convoluted cleavage and intergranular fracture, although the grain boundary decohesion resulted only in very shallow cervixes. (A similar fracture mode was observed also on thin tungsten wires after a vacuum anneal between 1300 and 2300°C at sufficiently low oxygen partial pressures [10,11].)

The microstructure had the following features after anneals at temperatures between 1300 and 2300°C.

- The average dislocation density has been estimated to be 10^{10} cm^{-2} [27]. (To this end a dedicated high-resolution X-ray diffractometer was applied with a suitable line profile analysis [28-30].)
- The contrast of the grain boundaries on transmission electron micrographs was typical for equilibrium grain boundaries [31].
- The first order internal stresses should relax upon a heat treatment at 1800°C for 1 hour, as was evidenced upon the annealing of wires twisted at the ambient [32].
- The grain structure was fibrous [9].
- The cross section of the fibres (delineated by high and low angle grain boundaries) is nearly equiaxial according to the transversal transmission electron micrographs [33].

We may therefore conclude: the microstructure of the samples had the inevitable features of a primary recrystallized microstructure, although the grain aspect ratio was high. The latter less common feature is due to the effect of potassium bubble rows on the process of continuous primary recrystallization. (This process is referred to also as microstructural coarsening [34].)

The recrystallized samples were split-free and remained split-free, when they were subjected to cyclic torsion with a true shear strain amplitude of 0.01 for 500 cycles [17]. (Thus, there were no latent splits [12] in our samples.)

The grain structure was revealed by the grain boundary grooves formed via thermal etching during annealing (Fig.1). The grain structure observed on the wire surface is quite similar to the structure that has been observed on scanning electron micrographs taken on longitudinal cross sections of similar samples [9]. (The grooves were due to thermal etching also in [9])

BENDING TEST

Figure 1 presents typical micro-crack configurations on the top of a bent sample where the wire is stretched. Evidently, the frequency of the micro-cracks was not governed alone through the following parameters of the grain structure:

- extent and shape of grain boundary segments between triple lines,
- position with respect to other grain boundary segments and
- position with respect to the actual axial direction of the wire.

This finding supports the view that both the load related stresses and the first order internal stresses will play only a secondary role in the micro-crack nucleation, as the most important component of both stress tensors is parallel to the actual axial direction of the wire, according to the simplest approaches of the elastic-plastic bending of cylindrical bodies. (This argument is valid also for the eventual circumferential (internal) stresses.)

What about the “hidden” parameters that should explain the observed “stochastic” nature of microcrack distribution? One “hidden” parameter is definitely the decohesion anisotropy of the grain boundaries [35]. In other words this means: the crystallographic parameters of the considered grain boundary segment have a marked effect on the magnitude of tensile traction that induces the boundary decohesion. A second effect of equal importance is exerted by the compatibility stress. This statement describes the following scenario. The average traction, acting on a short grain boundary segment, will be effected not only by the load related stresses and the first order internal stresses, but also the second and third order stresses will have their own effect. The latter kinds of stresses are due to the plastic mismatch between neighbouring grains, since misoriented grains display different plastic compliance with respect to both the external load and to the first order internal stresses. The reactive internal stresses accommodating the said plastic mismatch (the so called compatibility stresses) are the primary manifestation of grain interaction. They will vary also within a single elongated grain. In addition, the compatibility stresses may be quite high in the vicinity of grain boundary triple lines, as it has been recently evidenced by means of micro-hardness measurements on polycrystalline molybdenum [36]. Of course, the incompatibility stresses may have a marked variation also on longitudinal grain boundary segments in certain cases. Typical examples are the kinkled longitudinal grain boundaries, or certain curved grain boundary segments, the curvature of which is stabilised by the pinning effect of the potassium bubbles. The importance of such features is well supported by the studies of Fujii and co-workers on AKS tungsten [37]. They observed very different fracture behaviour on wires with different types of interlocking grain structures that were produced through the application of a broad range of heating rates of the anneals.

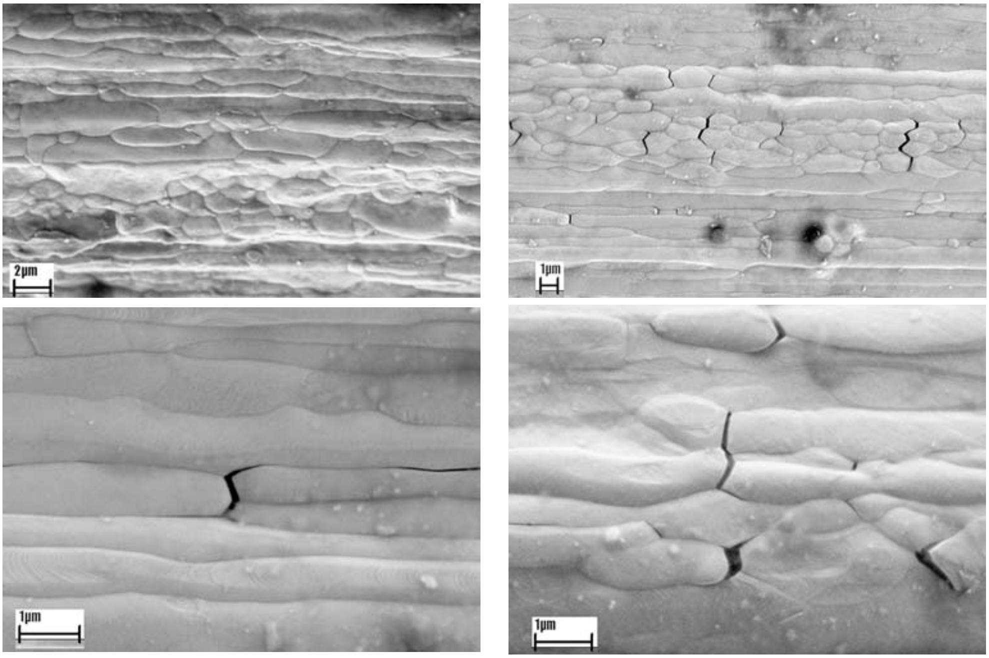

Fig.1. Micrographs taken on the top of a bent recrystallized wire revealing large micro-crack free areas and typical positions of micro-cracks.

We may therefore conclude according to Fig.1, a great number of longitudinal microcracks arise from the stress field generated by short, non-longitudinal grain boundary segments that have been opened up under the action of the compatibility stresses arising from the triple line effect.

The studied samples were free from long longitudinal macro-splits. This feature may be taken as a piece of evidence that the extent of applied strain was too low to generate macro-splits in a sufficiently soft microstructure. Since the leading macro-splits are longitudinal in the bent as-drawn wires, let us stress that the typical transversal micro-cracks are able to induce splitting along the longitudinal grain boundaries through their stress field, induced by the external stress and the first order internal stresses. However, it is easy to realise that the nucleation of longitudinal cracks will be a relatively rare event, which requires also the support of internal stresses. The essential point is that the nucleation of long longitudinal cracks has two prerequisites: (i) the transversal micro-crack has to be sufficiently broad, and (b) it should join to a longitudinal grain boundary segment that has a relatively low critical de-bonding traction. These prerequisites may exert a decisive effect on the frequency of the macro-splits, and this frequency may remain low with respect to the frequency of micro-cracks when the compatibility stresses are low. This kind of argument was also successful in the explanation of the low frequency of macro-split formed upon free-end torsion in samples where the micro-structure of which was virtually identical with the one of the present samples [17].

GRAIN BOUNDARY DECOHESION DURING NECKING

This work intends to ascribe the formation of longitudinal splits upon necking in the tensile test to the typical dislocation structure of heavily drawn and stress relieved wires. Therefore, let us first summarise the items of information on this topic. The transversal transmission electron micrograph Fig.2a shows a relatively thick and wavy black contrast at the grain boundaries of as drawn tungsten [31,33,34], corresponding to a dense network of dislocations forming an envelope along the grain boundaries. (The micrograph is not sensitive to the misorientation of the boundaries. It is known, however, that at least 1/3 of the boundaries are high angle grain boundaries (θ >15°) [34].) Fig.2b depicts the fracture surface of a sample annealed at 600°C. A major part of the neighbouring splits caused knife edge fracture with a convoluted edge. The minor part of the neighbouring splits encloses islands of transversal fracture. With increasing annealing temperature, the area of the transversal islands increases. The fracture is cleavage dominated on samples annealed at 1300°C.

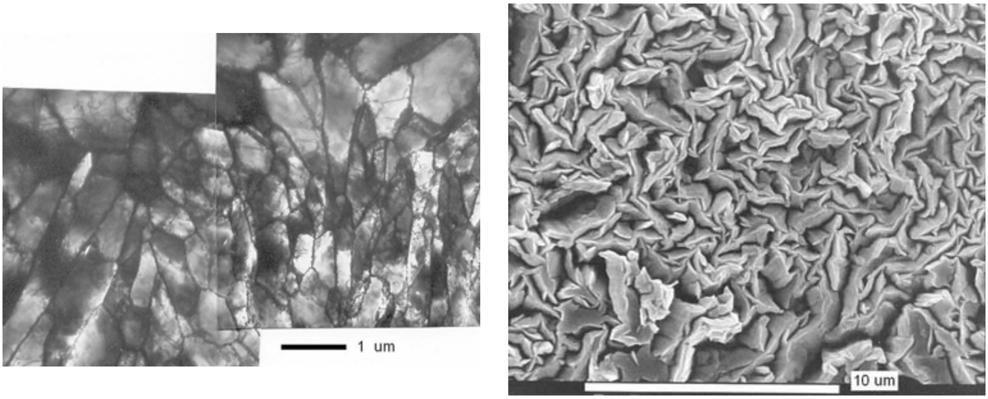

Fig.2. (a) Transversal TEM showing the dislocation envelope of (sub)grain boundaries, as well as the directional variation of the transversal long axis of the (sub)grains (Hosford structure). (b) The morphology of the transversal traces of the splits also reveals a Hosford-type grain structure. The majority of the fibres with opened up grain boundaries undergoes a knife edge fracture, while a small part breaks with transversal fracture along a finite cross section.

The asymmetric shape of the X-ray line profiles suggests [27,30,31] that the dislocation layer on both sides of a boundary consists of a network of polarised dislocation dipoles formed from dislocations that were not able to cut through the grain boundary during deformation. The density of dislocation in the envelope increases both with increasing wire drawing strain and decreasing drawing temperature, as expected [19,32-34].

The dislocation density in the dislocation envelopes of the grain boundaries may be as high as 10^{12} cm^{-2} in as-drawn commercial tungsten having a diameter of 173 μm [28,31]. Of course, the dislocation density in the envelopes decreases upon annealing, and longitudinal transmission electron micrographs reveal equilibrium grain boundaries without dislocation envelopes and extrinsic grain boundary dislocations with a low frequency even at relatively low annealing temperatures (Fig.3a) [31], although few grain boundary segments having a dislocation envelope persist with low frequencies up to annealing temperatures of about 1000 or 1200°C [31].

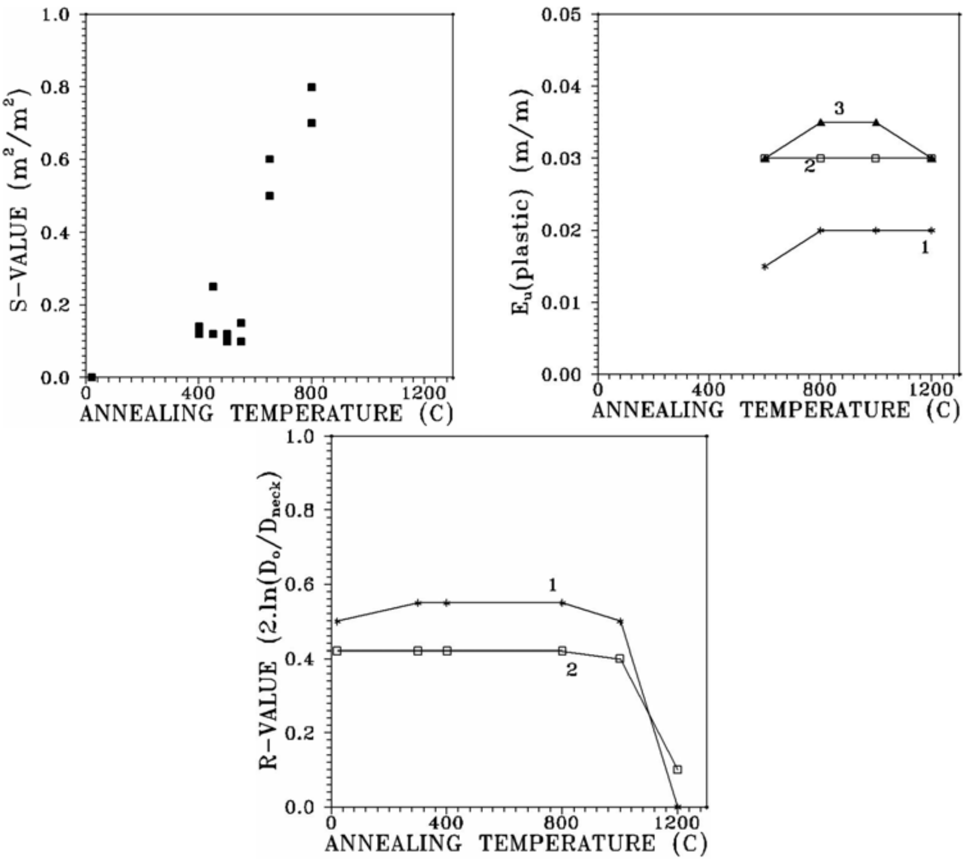

Fig.3. (a) The fraction of the equilibrium grain boundary area with respect to the total grain boundary area as determined on longitudinal TEMs (S-value) [31]. (b) The annealing curve of uniform plastic elongation, E_u(plastic), before necking in a tensile test at the ambient for three different proprietary processing routes. (c) The annealing curve of the R-value for two proprietary processing routes. (The annealing time was 15 min.).

Why may the dislocation envelope of grain boundaries play an important role in micro-crack nucleation? The scenario is as follows. When a grain boundary segment is opened up by grain boundary decohesion, then not only the energy of the grain boundary will be released in order to compensate for the energy of the new free surface

segments, but also a major part of the energy of the attached dislocation network will be released, because a great majority of the envelope dislocations will escape through the new free surface created by fracture. (The attractive interaction is very high among a segment of free surface and the dislocations that are close to it, and this fact favours the short-path glide.) Simple models show that the energy released per unite boundary area through dislocation, escape on the very same area, is close to the surface energy of the equilibrium high angle grain boundaries in commercial as drawn tungsten, having a diameter of 173 μm [27]. The possibility of such high energy release will, of course, lead to an easy grain boundary decohesion. On the other hand, when the grain boundaries are not covered with a dislocation envelope any more (Fig.3a), then the splitting is suppressed, the splitting supported necking is diminished, the R value becomes low (Fig.3c) and cleavage becomes the preferred mode of fracture.

Let us turn to the tensile test at the ambient. We have seen that a certain limited amount of uniform plastic elongation yields to simultaneous necking and splitting with high R values in as drawn samples. In contrast: a similar amount of uniform plastic elongation results in cleavage dominated fracture in primary recrystallized samples. In this case, the transgranular cervices are shallow and the necking is quite limited (low R-values). This transition in necking behaviour takes place when the samples are annealed at temperatures higher than 1300°C. Although this transition is not connected with any marked change in the flow stress or in the mean transversal grain intercept, it essential features (Fig.3) can be easily interpreted in terms of the following scenario. The grain boundary area with dislocation envelope decreases with an increasing annealing temperature. A simultaneous change takes place also in fracture: the total area of transversal fracture islands increases with an increasing annealing temperature (Fig.2b) It is, however, quite remarkable that the limited increase in the total area of the transversal fracture islands below 800°C does not markedly effect the extent of necking (Fig.3c). The rationalisation of this interesting question is outside the scope of the present work. However, when the dislocation envelope of the grain boundaries is annihilated to a critical extent, then also the splitting of the grain boundaries diminishes and the splitting supported extended necking ceases.

DISCUSSION

The results of free-end torsion at the ambient are in excellent agreement with the interpretation of the results of the present work. In this context, one should stress the following points. Samples prepared from the same stock and in the same way as the samples of the presented work showed the following behaviour.

- The deformation until fracture was very high, the true shear strain at the surface amounted, γ to 2.
- Although the longitudinal opened up by decohesion at very low strains ($\gamma = 0.01$) in as drawn and stress relieved samples (having an annealing temperature lower than 1200°C), the grain boundary splitting started at $\gamma = 0.1$, when the samples were annealed at 1800°C.
- The frequency of the macro-splits was very low with respect to that of the nearly transversal micro-cracks.
- The microcracks had the same stochastic nature as observed in the bent samples of the present study.
- The first order circumferential internal stresses due to wire drawing and free-end torsion seem to support the propagation of longitudinal macro-splits.

One may analyse also the splitting during wire drawing along similar lines. Of course one has to take into account that the stress field of the dislocation envelope on a certain segment of a grain boundary depends on a great number of parameters which have not been discussed in the present work (local variation of the surface loading due to local variation of lubrication etc.). One may not even exclude that the average traction of the compatibility stress will have a marked range of variations even on the quasi longitudinal boundaries. In addition, the circumferential internal stress [18,19] may have a marked effect on the propagation of macro-splits, although their fluctuation along the wire length may also effect the formation of longitudinal micro-cracks. The observed sensitivity of splitting to the processing parameters [18,19] can be also rationalized in terms of the recovery of the dislocation envelope of the grain boundaries. The rate of this process is similar to the rate of polygonization that has been evoked to the qualitative rationalisation of the marked effect that the preheating temperature of drawing exerts on the split propensity [19,20,26].

CONCLUSIONS

1. Transversal, short and non-propagating micro-cracks evolve upon bending with marked frequency.
2. The micro-cracks may support the nucleation of longitudinal macro-cracks through their stress field.
3. The dislocation envelope on the grain boundaries supports grain boundary decohesion. Its partial relaxation may have a beneficial effect on the split propensity.

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