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THE OCCURRENCE AND CHARACTERISTICS OF LÜDERS DEFORMATION IN POLYCRYSTALLINE ZINC

WYSTĘPOWANIE I CHARAKTERYSTYKA DEFORMACJI LÜDERSA W POLIKRYSTALICZNYM CYNKU

After some "special" low-temperature rolling and subsequent ageing at room temperature of polycrystalline zinc of 99.99% purity L ü d e r s-type deformation occurs. This effect results from the localisation of deformation (twinning) which leads to "asynchronization" of work hardening, recovery and recrystallization processes in different parts of material. As a consequence, polygonal substructure and, probably, "special" grain size are formed. L ü d e r s strain increases with decreasing grain size and deformation rate as well as with the increase of deformation temperature.

W cynku o czystości 99.99%, poddanym „specjalnej” procedurze walcowania w niskiej temperaturze a następnie leżakowaniu w temperaturze otoczenia, występuje deformacja typu L ü d e r s a. Efekt ten jest rezultatem lokalizacji deformacji (bliźniakowanie), powodującej „rozsynchronizowanie” procesów umocnienia, zdrowienia i rekrytalizacji w różnych obszarach materiału. Prowadzi to do formowania poligonalnej podstruktury i, prawdopodobnie, „specjalnych” granic ziaren. Wielkość deformacji L ü d e r s a rośnie wraz ze zmniejszeniem wielkości ziarna i prędkości deformacji a także ze wzrostem temperatury deformacji.

1. Introduction

The occurrence of L ü d e r s-type deformation is usually associated with one of the two metal structures: i) the presence of alloy addition or ii) instability of the existing dislocation substructure, resulting from "intrinsic" or "induced" change of

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the deformation mechanism [1, 2]. In each case the initiation (or continuation) of plastic flow requires definitively higher stress than its propagation in a narrow zone (L ü d e r s band).

The unexpected appearance of such form of deformation localisation in pure (99.99%) aluminium [3, 4] prompted the authors to look for its origin in the absence of a sufficient number of dislocation sources in strongly polygonized structure [3] or in the domination of "special" grain boundaries with non-equilibrium structure, being the result of applied procedure of sample preparation (in particular the low-temperature discontinuous recrystallization). These "special" grain boundaries require high stresses for the initiation of the "hard" dislocation sources and favour the formation of stable dislocation pile-ups [4—7].

The second concept has been also proposed to explain the absence of any relation between the occurrence of L ü d e r s phenomenon and the grain size in CuSn2% alloy recrystallized at the temperature 723 K ($0.53 T_m$). However, a careful analysis of the presented microstructures of rolled sheet evidently indicates the existence of a close relation between this phenomenon and the tendency toward structure heterogenization during rolling and the conditions of subsequent recrystallization. Moreover, it seems to be closely related with the "scale" of the localisation phenomenon and the volume fraction of the areas of localised deformation. Localisation of deformation in the "micro-" scale (twinning) leads to the appearance of fairly small "plateau" on the tensile curves. In turn, the onset of "macroscopic" localisation of deformation in the shear bands is responsible for the occurrence of both yield point and the tensile force instability, typical for L ü d e r s strain.

Thus, the question may be asked, in what way the non-homogeneous deformation of rolled sheet metal is "inherited" by the recrystallized material as a tendency toward localised flow in L ü d e r s band?

Both the twins and the shear bands are work hardened areas in relation to the surrounding matrix. The first ones — as a result of twinning transformation of the slip dislocation into sessile dislocation [9—15]; the second ones — due to the engagement of a great number of dislocations in the local deformation [16, 17]. Work hardening by twinning has been estimated (on the basis of hardness test and the critical resolved shear stress measurements) to be 2—3-fold [12—15], whereas the inside area of a shear band shows at least 1.3 times higher microhardness than that outside the band [16].

Hence the fact (which in the case of twinning has been known for a long time) that these areas are particularly susceptible to static recrystallization is not surprising. S c h m i d and B o a s [14] has demonstrated that recrystallization in a twinned area of Cd single crystal occurred already after 1 minute at the temperature 418 K, whereas the remaining part of the crystal did not become recrystallized until the temperature exceeded 513 K. Similar effects of the "acceleration" of recrystallization were observed in the shear bands areas both in single- and polycrystals [17, 18].

Thus, the localisation of deformation is responsible for the “asynchronous” recrystallization of the different areas of material, especially during low-temperature annealing. In this fact, as it seems, one should look for the reason of the formation of the grains of energetically differentiated boundaries. It can be assumed that it is just these grains which nucleate outside the areas of localised deformation, that have a definitely less perfect (“special”) grain boundaries and that they will determine the quality of the “hard” dislocation sources [5—7]. The fact of the decay of L ü d e r s deformation in sheet metal with too small degree of structure heterogenization (as a result of twinning), as well as in the case of structure saturation by the shear bands [8] seems confirm the correctness of the presented reasoning.

Polycrystalline zinc has been selected for experimental verification of this concept. As it is known, the structure of zinc sheet and its mechanical properties depend on the degree of purity and the applied technology, in particular: temperature and intensity of rolling (reduction in a single rolling pass, cooling between the passes, ageing conditions between the passes and after completion of rolling). This enables to control relatively easily the temperature of rolled band, even to maintain it at a level close to ambient temperature ($0.42 T_m$), and in this way to control both the degree of structure heterogenization (as a result of twinning) and the processes of structure regeneration (recovery and/or recrystallization).

To prove the existence of a relation between structure heterogenization (through predeformation), conditions of its regeneration and the occurrence of L ü d e r s deformation is the objective of present study.

2. Experimental

Zinc of 99.9925% purity, containing of admixtures (in wt.%) in the following amounts: Fe 0.0029%, Pb 0.0017%, Cd 0.0018%, Cu 0.00093% and Sn 0.0016% was used. Vertically cast ingots of the zinc, measuring $9.8 \times 20 \times 100$ mm were subjected to unidirectional rolling up to 95% reduction. The rolling was conducted according to the three various procedures differing in the intensity of process and the thermal effects. The ratio L/H_o (where L is the length of the rolling gap, and H_o is the thickness of the sheet before the successive pass) was about 1, 2 and 3, respectively. After each pass the rolled band was cooled with a flow of air, and, as a result, its temperature did not exceed 320, 335 and 350 K.

Material prepared in this way was subjected to structural observations and mechanical tests immediately after rolling, after ageing at 298 K and after annealing at 373 K. The tensile test was carried out on samples with the gauge length 10×25 mm in the range of temperature and deformation rate; 173—373 K and 3.3×10^{-5} up to $1.3 \times 10^{-2} \text{ s}^{-1}$, respectively. For each testing conditions at least 5 samples were tested. Besides the force-elongation record, for part of the samples subjected to tension at ambient temperature, there was also recorded the local elongation using extensometer of 6 mm basis.

3. Results

3.1. Conditions of the occurrence of Lüders deformation

Immediately after rolling all tensile curves of zinc have a parabolic character, but their course becomes differentiated with the time of ageing (Fig. 1). Application of the two rolling procedures results in stable or almost stable mechanical properties of the material and the sheet structure is then either typical for the development of

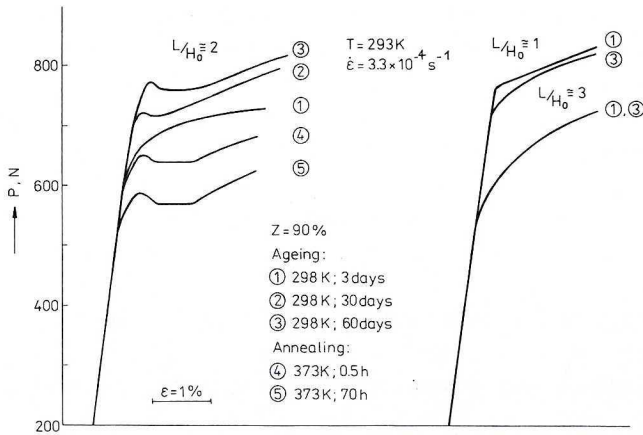


Fig. 1. The influence of rolling procedure and of subsequent ageing or annealing conditions on the course of tensile curves. Rolling reduction 90%

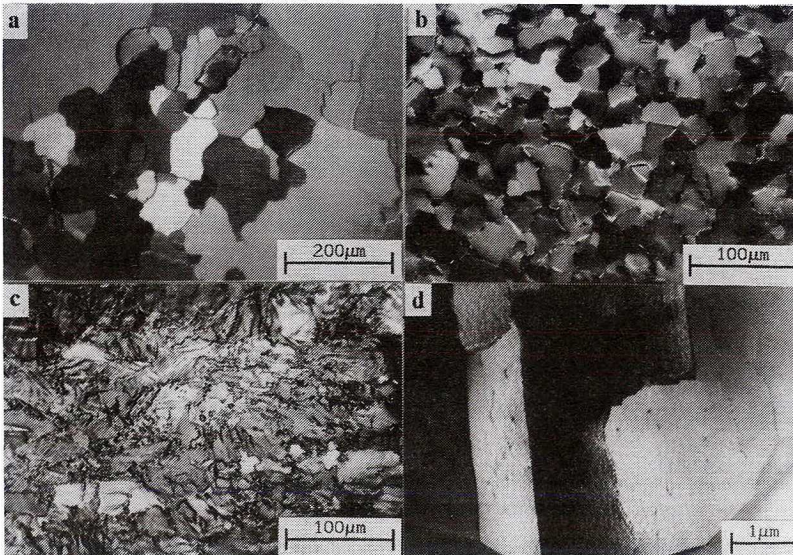


Fig. 2. The structure of zinc sheet after ageing at ambient temperature during 60 days. Rolling reduction 90%. a) $L/H_0 \cong 3$; b, d) $L/H_0 \cong 2$; c) $L/H_0 \cong 1$

secondary recrystallization ($L/H_o \cong 3$) or it shows the features of a mixed structure, in which the areas with fine, recrystallized grains are formed in the neighbourhood of the primary grains ($L/H_o \cong 1$) (Figs. 1, 2 a, c).

The yield stress of a sheet rolled with medium intensity ($L/H_o \cong 2$) significantly increases with the ageing time, and the initiation of plastic flow is then accompanied by distinct mechanical instability (Fig. 1). In these sheets there can be observed fine, almost equiaxial grains, and a considerable part of them reveals the presence of polygonal subgrains (Fig. 2 b, d). The stability of this structure is responsible for the fact that the mechanical effect is permanent — it is not changed by a few years ageing at ambient temperature, and annealing at temperature not higher than 373 K causes only a reduction of the yield stress (Fig. 1).

Examination of the texture has shown that in zinc, demonstrating L ü d e r s effect, besides the standard texture component $[0001] \pm \alpha(\text{ND-RD})$ there appears the

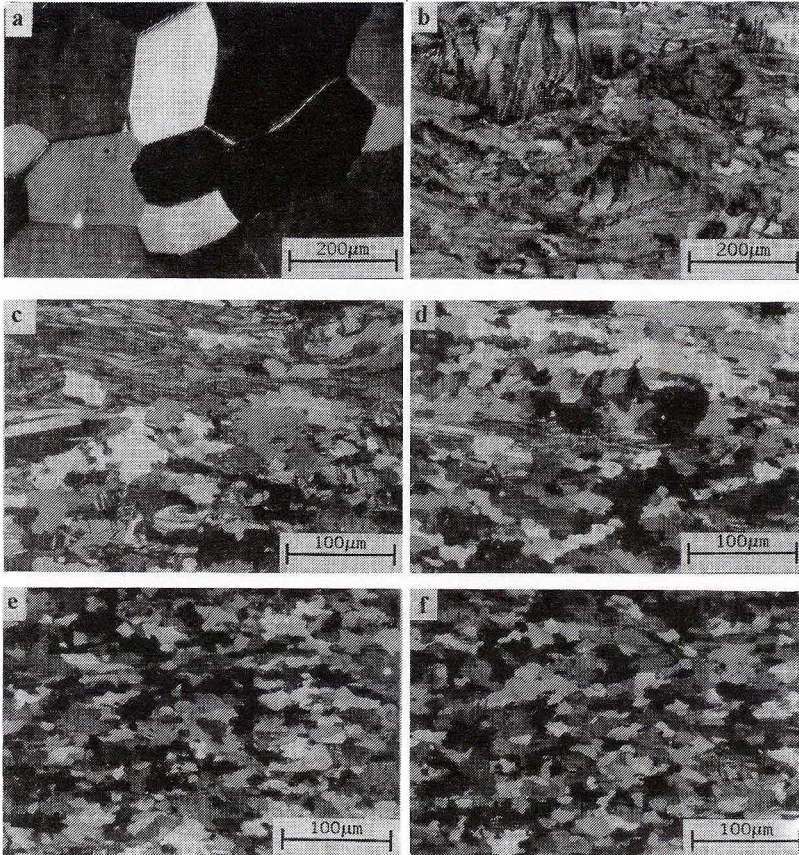


Fig. 3. The structure of zinc: a) ingot, b-f) sheet, observed immediately after rolling. Rolling reduction: b) 50%; c) 70%; d) 80%; e) 90%; f) 95%. $L/H_o \cong 2$

significant component $[0001] \parallel \text{TD}$, which is usually absent and whose contribution has been estimated at about 15% [19].

The results suggest, that the occurrence of yield point is determined by the development of zinc structure in the course of rolling (Fig. 3). The coarse-grained structure undergoes strong heterogenization and fragmentation as a result of two processes proceeding in parallel; i) twinning and ii) static recrystallization occurring mainly during the breaks between rolling passes. The consequence of the latter is the formation of fine-grained structure of "new" grains both in some of the earlier formed twins and in areas adjacent to the "old" grain boundaries.

The process of structure heterogenization proceeds up to rolled reduction of 70%. (Fig. 3 a-c). The effect of ageing at ambient temperatures (and even annealing at 373 K) is retained in the structure of the primary twinned grains, and, as a result, the final mixed structure resembles that presented in Fig. 2c. The stretched samples show then a distinctly marked yield stress (the extensometer record suggests homogeneous deformation) and the heterogenization of structure has as result a great scatter of the yield stress value (Fig. 4).

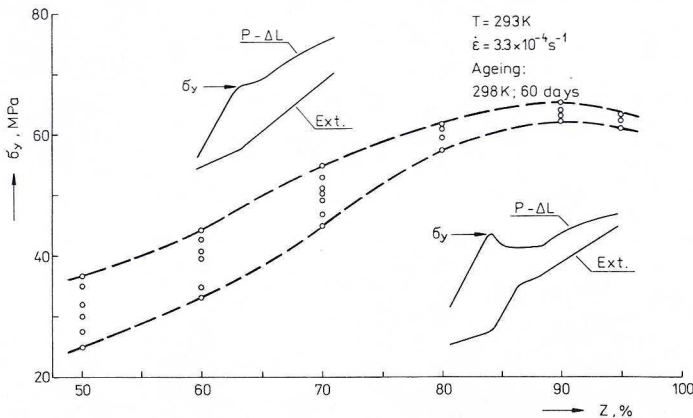


Fig. 4. The influence of rolling reduction on the yield stress of zinc sheet. $L/H_0 \cong 2$

In sheet deformed to 80% at least one can observe the domination of "new" grains of elongated shape and a residual presence of the deformation structure — "old" grains with the twin bands situated almost parallel to the sheet surface (Fig. 3 d-f). During ageing at ambient temperature the structure of zinc significantly changes and an almost equiaxial grains are formed (Fig. 2 b). The final size of grains diminish from about 25 μm to about 10 μm with increase of the predeformation from 80 to 95%.

3.2. Characteristics of Lüders deformation

The influence of temperature and deformation rate on the initial stage of the tensile curves and the upper yield stress as well as the value of Lüders strain (ϵ_L) is

shown in Figs. 5 and 6. At the temperatures 298 K and 373 K L ü d e r s effect occurs in a wide range of deformation rate (Fig. 5). We can observe the standard

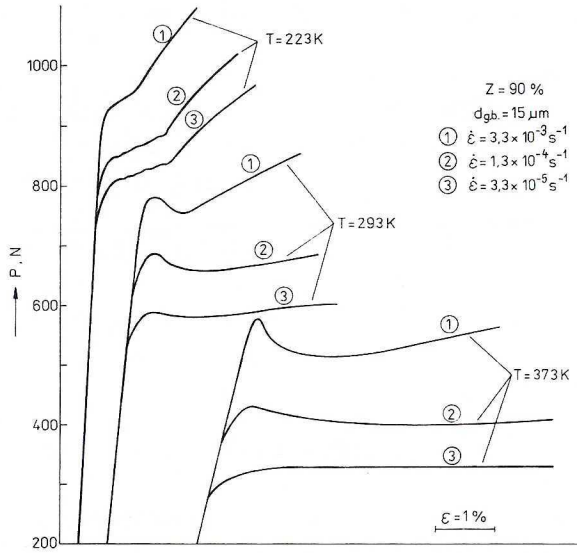


Fig. 5. The initial part of tensile curves of zinc obtained at different temperatures and strain rates. $L/H_0 \cong 2$

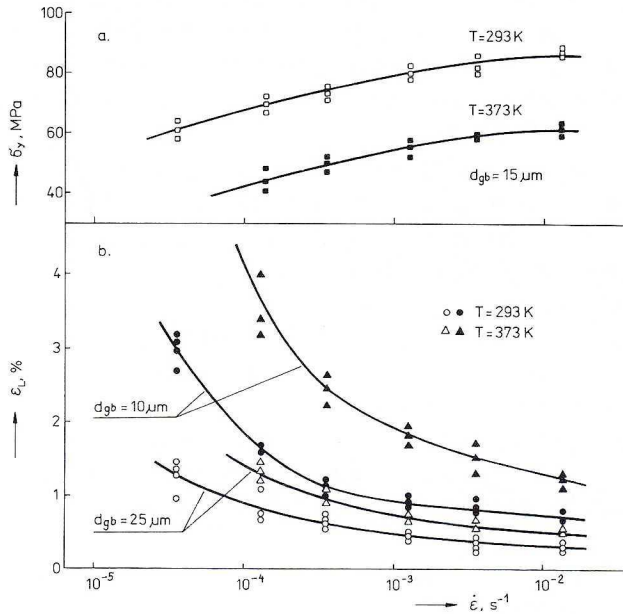


Fig. 6. The influence of grain size, deformation temperature and strain rate on the upper yield stress (a) and on the Lüders deformation (b) in zinc. $L/H_0 \cong 2$

behaviour of the upper yield stress, that is decrease with increasing temperature and reduction of the deformation rate. The value of ε_L is distinctly higher for a more fine-grained structure, but also definitely greater for higher temperature and lower deformation rate (Fig. 5, 6). Worthy of note is the "flat" course of tensile curves for samples tested at the temperature 373 K at the lowest rate $3.3 \times 10^{-5} \text{ s}^{-1}$. Under such conditions the material demonstrates extended plasticity (about 200%) and the strain rate sensitivity ($m = d \ln \sigma / d \ln \dot{\varepsilon}$) exceeds 0.2. The width of L ü d e r s band at the moment of nucleation at ambient temperature equals about 3—4 mm.

At lowered temperatures there take place a gradual transition to homogeneous deformation. Still at 223 K the increase of the tensile force is associated with its rather small oscillations what means that localisation of deformation in L ü d e r s band is accompanied by simultaneous homogeneous deformation of the remaining parts of the sample (Fig. 5). All the tensile curves from the temperature 173 K are "smooth" and parabolic.

4. Discussion

In the first part of the study it has been demonstrated that a necessary condition for the appearance of L ü d e r s deformation in pure zinc is to obtain fine-grained structure with (almost) equiaxial grains, with a great proportion of those containing polygonal boundaries. This condition can be satisfied by a proper selection of the rolling parameters, sufficiently high predeformation and sufficiently long time of ageing at ambient temperature. However, a simple refinement of the structure is not responsible for this effect, since on many occasions zinc of definitively smaller grains has been obtained which in the tensile test become deformed in a homogeneous mode.

Observations of the zinc structure shown evident lack of "synchronization" of the processes of the strain hardening, static recrystallization of a part of the structure and its renewed plastic deformation (with differentiated degree of reduction), and finally, of low temperature recrystallization and/or recovery in various volumes of the material (Fig. 3 b-d).

As a result of such a procedure a structure is formed which is composed of three groups of grains of different "history" and, as a consequence, of different substructure, as well as most probably, of different structure of grain boundaries:

- recrystallized grains, formed in the early stages of rolling, undergoing considerable plastic deformation (resulting in the elongation) (Fig. 3 b, c);
- grains, which were formed in the latter deformation stages (Fig. 3 c, d), undergoing a rather small plastic deformation, and
- grains, the formation of which as the last ones, takes place in the remaining areas of the "old" twinned grains (Fig. 3 d-f).

For among these grains, the first ones undergo most probably static recrystallization, whereas in the two remaining ones the recovery processes must be of great

importance. Since the simultaneous decay of the remainder of the deformation structure and formation of equiaxial grain is accompanied by the increase of the yield stress and the appearance of L ü d e r s strain one can believe that this effect is mainly the result of the processes of polygonization (Fig. 2 d); it can not be excluded however, that it is reinforced by the structural differentiation of the grain boundaries.

It is worth noticing, that usually all these factors which increase the upper yield stress of the material lead to the increase of the L ü d e r s strain; such behaviour is typical for alloys having the structure of both substitutional and interstitial solid solution [1, 20—22]. On the other hand, in zinc (similarly as in aluminium [4]), the value of ε_L , although increasing with decreasing grain size, surprisingly increases with the increase of the deformation temperature. Moreover, only in zinc, it increases strongly with the decrease of the deformation rate (Fig. 6). In turn, in some intermetallics, the athermal dependence of the upper yield stress is associated with a significant increase or with constant value of ε_L , as in Zr_3Al [23] or in Ni_3Al [24], respectively.

The discussed phenomenon takes place only in zinc of higher purity. Similar behaviour has not been observed either for zinc of commercial purity which excludes any relation with the ageing processes (in particular release of excess amount of Fe) or for zinc with small amounts of alloy addition such as, alternatively, Fe, Cu and Pb.

5. Conclusions

1. L ü d e r s deformation occurs in pure zinc of the grain size 10—25 μm in the temperature range from 293 K to 373 K, and in a wide range of deformation rate. The value of L ü d e r s strain increases with diminishing grain size and deformation rate and with the increase of the deformation temperature.

2. Obtaining a structure demonstrating the tendency toward L ü d e r s-type localisation requires appropriate rolling procedure, in particular maintaining low-temperature of the rolling band, application of sufficiently high degree of deformation (80% at least) and sufficiently long time of ageing at ambient temperature.

3. The structural cause of the occurrence of L ü d e r s effect is the localisation of deformation during rolling (twinning) which leads to heterogenization of structure and, as a consequence, to “asynchronization” of the processes of work hardening, low temperature static recrystallization and static recovery in the different areas of the material. The result of such a progress of the processes of structure regeneration is the presence of polygonal substructure and, probably, formation of “special” grain boundaries.

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