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## DEFORMATION INDUCED VACANCIES AND WORK HARDENING OF METALS

## WAKANCJE INDUKOWANE ODKSZTAŁCENIOWO A UMOCNIE NIE METALI

The paper follows a previous one on effects of non-equilibrium vacancies on strengthening [1] by focussing on the formation mechanisms of vacancies and/or vacancy agglomerates through plastic deformation, and on their influences to both work hardening after deformation ('static effects'), and during it ('dynamic effects'). One has also to distinguish between (i) *direct* (and (ii) *indirect* effects of vacancies/agglomerates to hardening characteristics. While at *small strain* the *direct* interaction of vacancies/vacancy agglomerates with dislocations governs the macroscopic hardening characteristics, at *large strains* the *indirect* effect of these vacancies/agglomerates prevail; here, the growing number of dislocations acts as sinks for the vacancies/agglomerates which, however, induce enhanced climb and annihilation of edge dislocations leading to marked softening effects even at low deformation temperature  $T \approx 0.2 T_m$  ( $T_m$  is the melting temperature in K). Examples are given for effect (i) where both the critical resolved shear stress  $\tau_c$  and the hardening coefficient  $d\tau/d\gamma$  were found to change up to 20% in hcp — metals and alloys, but still markedly in fcc ones. Effects of type (ii) are shown to typically rule the so-called stage *V* of deformation at low deformation temperatures, for dynamic but also static cases. With the latter, additional annihilation of edge dislocations will occur during unloading which can lead to an absolute decrease of dislocation density and thus, of macroscopic strength. Consequently, iterative modes of deformation such as rolling, extrusion and wire drawing are predestined to show this effect. Both the direct as well as the indirect effect of deformation induced vacancies deserve particular interest in cases where conventional large strain deformation modes are combined with hydrostatic pressure in order to produce ultrafine grained or nanocrystalline metals. These materials exhibit outstanding physical properties, e.g. a strength being up to a factor 3 higher than the same material with coarse grains. The hydrostatic pressure is thought to restrict diffusion via vacancies which should allow for higher accumulation of both deformation induced vacancies and/or dislocations. First experimental results are presented which suggest the extra hardening to arise mainly from vacancies or vacancy agglomerates, in the same of a direct interaction with dislocations as defined above.

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Prezentowana praca jest wynikiem wcześniejszych obserwacji efektów wywołanych wpływem nierównowagowych wakancji na umocnienie [1]; dotyczy mechanizmów tworzenia się wakancji i/lub ich aglomeratów w czasie odkształcenia plastycznego i ich wpływu na umocnienie po deformacji ('efekt statyczny') i podczas deformacji ('efekt dynamiczny'). Praca dotyczy także odróżnienia (i) *bezpośredniego* i (ii) *pośredniego* efektu wpływu wakancji/aglomeratów na charakterystykę umocnieniową. Podczas, gdy *małe odkształcenia* prowadzą do *bezpośredniego* oddziaływania wakancji/ich aglomeratów z dyslokacjami i powodują makroskopowe efekty umocnienia, to *duże odkształcenia* powodują *pośrednie* efekty od wakancji/lub aglomeratów; prowadzą do wzrostu ilości dyslokacji i procesów zanikania wakancji/aglomeratów, jak również do wzrostu wspinania i anihilacji dyslokacji krawędziowych, powodując efekty zdrowienia, nawet w niskich temperaturach deformacji  $T \approx 0,2 T_m$  ( $T_m$  jest temperaturą topności w K). Efekty takie obserwowane są (i) dla metali i stopów o strukturze HZ, — gdzie następuje efekt wzrostu do 20% oraz mniej wyraźny dla struktur RSC, — krytycznego naprężenia ścinającego  $\tau_c$  i współczynnika umocnienia  $d\tau/d\gamma$ . Efekty typu (ii) są obserwowane jako typowe w czasie odkształcenia w tzw. *V* stadium deformacji w niskich temperaturach tak dla dynamicznego jak i statycznego przypadku. Następuje potem dodatkowa anihilacja dyslokacji krawędziowych prowadząca do spadku wytrzymałości, to może doprowadzić do całkowitego spadku gęstości dyslokacji i spadku makroskopowego umocnienia. Powtarzająca się deformacja poprzez walcowanie, wyciskanie, czy ciągnięcie predestynuje obserwowany efekt. W obu przypadkach, pośredni i bezpośredni efekt deformacji indukującej wakancje, zasługuje na szczególną uwagę w przypadku, gdy konwencjonalne duże odkształcenia są połączone z naprężeniem hydrostatycznym i prowadzą do otrzymania ultradrobnoziarnistej struktury lub metali nanokrystalicznych. Tak otrzymane materiały wykazują wybitne własności fizyczne, np. wytrzymałość do 3 razy wyższą niż takie same materiały o grubym ziarnie. Wydaje się, że ciśnienie hydrostatyczne ogranicza dyfuzję poprzez wakancje, ale pozwala na osiągnięcie wyższej koncentracji wakancji i/oraz dyslokacji indukowanych odkształceniem. Prezentowane wyniki eksperymentalne wskazują, że dodatkowe umocnienie wywołane jest głównie wykancjami lub aglomeratami wakancji, które mają wpływ na bezpośrednie oddziaływanie z dyslokacjami.

## 1. Generation of vacancies by plastic deformation <sup>\*)</sup>

It has been clear since four decades that plastic deformation induces not only the formation of dislocations but also of vacancies and vacancy agglomerates. Although originally even interstitial atoms have been claimed to be generated by deformation [2], theoretical as well as more recent experimental evidence does not support this opinion: this is the high mobility of interstitial atoms compared to vacancies on the one hand, and the results of nuclear methods (Mößbauer [3] and PAC [4] spectroscopy) on the other which showed no presence of interstitial atoms like in irradiated samples during isochronal annealing treatment. Direct observations of

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as-deformed single crystals by TEM in hcp metals by Mikulowski et al. [5] and in fcc metals recently published by Dai and Victoria [6] clearly revealed the presence of vacancy-type agglomerates after plastic deformation.

Two models have been suggested for the deformation induced generation of vacancies: one is the so-called "jog moving" mechanism suggested by van Bueren [7, 8]: This states the cutting of screw forest dislocations by screw dislocations moving in the slip plane, generating dislocation jogs in these dislocations which generates the higher densities of vacancies, the higher is the angle of these dislocation lines related to their Burgers vector (Fig. 1). According to this concept, the vacancy concentration increases by  $c_v = (N_f/36) b L \gamma$  in deformation stage I, and  $c_v \cong 0.03 \gamma^2$  for stage II, where  $N_f$  is the forest dislocation density,  $b$  is the Burgers vector of moving dislocations,  $L$  is its mean free path, and  $\gamma$  is the resolved shear strain. In what concerns stage I, the experimentally measured vacancy concentrations are in between  $10^{-5}$  and  $10^{-6}$  which is shown in Fig. 2 as a result from calorimetric measurement. Vacancy densities can be measured through constant rate heating by special low temperature inert calorimeters [10, 11] evaluating the area under the first heat flux peak arising in deformed samples (Fig. 3). The measured vacancy concentration only agrees with the theoretical one if the forest dislocation densities are assumed to be  $10^9 \text{ m}^{-2}$  or higher. However, no such high forest dislocation densities have been found by experiment particularly for hcp metals [12]. Moreover, the idea of moving jog creation of vacancies strongly fails in stage II

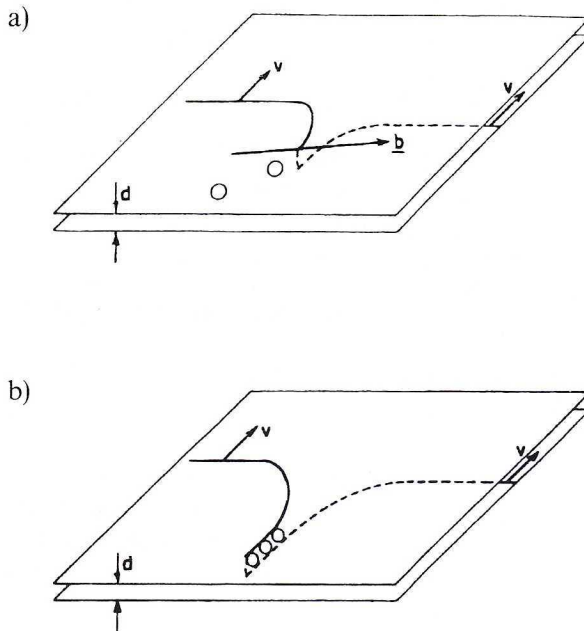


Fig. 1. Mechanism of "jog dragging (- moving)" for production of vacancies in the slip plane (van Bueren [7, 8], from [9])

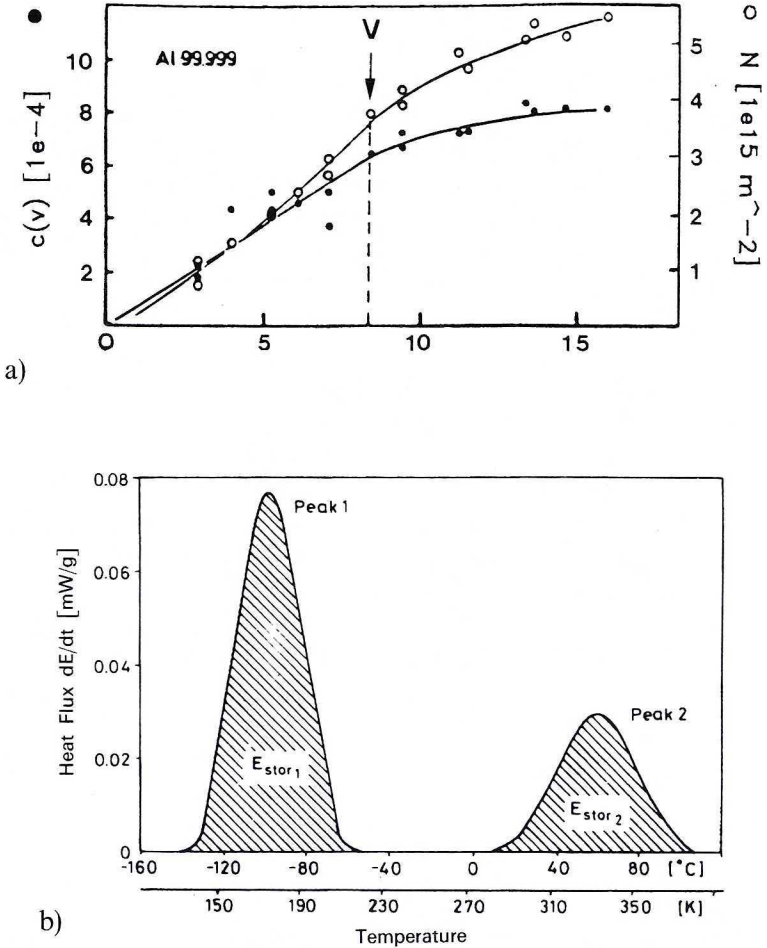


Fig. 2. a) Measurement of density of deformation induced vacancies and dislocations in large strain deformed Al 5N polycrystals (from [10]), b) Calorimetric measurements of stored defect energies in deformed Zn 4N single crystals using the area under exotherm peaks being the measures for density of vacancies (peak 1) and dislocations (peak 2) (from [11])

where it predicts vacancy concentrations being two orders of magnitude too high compared to experiment, although the parabolic increase of vacancy concentration with strain is reflected well [13].

The second model has been suggested by S a a d a [14] which has been based on idea of Hirsch [15] that the moving slip dislocations meet an repulsing dislocation tree, bow out and meet on neighbouring slip planes forming a row of vacancies in between its two dislocation branches (Fig. 4). Then the vacancy concentration can be derived as

$$c = (\alpha_1 \cdot \alpha_3 / \alpha_2) \cdot 1/\mu \cdot \int_0^{\epsilon} \sigma d\epsilon, \tag{1}$$

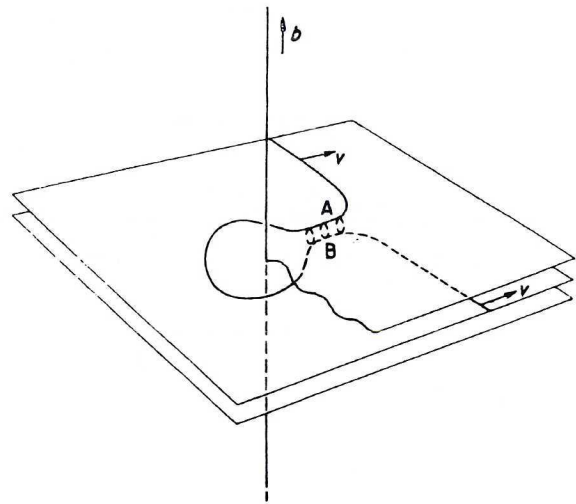


Fig. 3. Mechanism of production of deformation induced vacancies by dislocation annihilation (S a a d a and H i r s c h [14, 15], from [9])

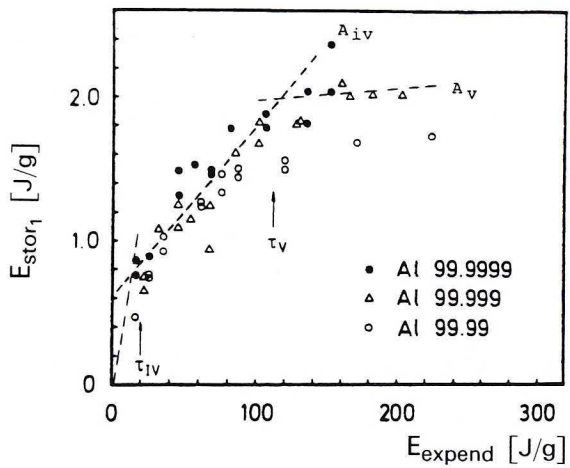


Fig. 4. Fraction of vacancy concentrations in terms of total expended deformation energy. The slope is reflected by the quantity A which shows a characteristic value for each deformation stage (from [17])

where  $\alpha_1$  rules the dependence of  $c$  on the difference of moving directions of dislocation branches and their B u r g e r s vector,  $\alpha_2$  is the coefficient of O r o w a n stress necessary for bowing out of slip dislocation between neighbouring dislocation trees, and  $\alpha_3$  gives the fraction of tree dislocations with respect to total dislocation density. Since  $\alpha_1 \approx \alpha_2 \approx 0.25$  [16] and  $\alpha_3 \approx 0.1$  at least at the beginning of deformation, the vacancy concentration can be calculated from actual shape of corresponding stress-strain relationships. A quite good coincidence of calculated

with experimental data can be stated, at least for stages I and II. In principle, this model would allow to calculate  $c$  for even higher deformations as long as the stress-strain characteristics is known; however, Saada's concept in its actual form does not yet account of dislocation and/or vacancy annihilation processes starting in stage III and/or stage V, respectively. Therefore it is not surprising that the product of the  $\alpha$ 's, being  $A \equiv (\alpha_1 \cdot \alpha_3 / \alpha_2)$  shows a fictitious decrease if one considers the experimentally measured vacancy concentrations  $c$  as a function of area under  $\sigma - \varepsilon$  relationships (Fig. 5): The slope representing the  $A$  — values amounts to  $A \approx 0.1$  for stage I and II, but slows down to  $A \approx 0.03$  in stage III, and to  $A \approx 0.015$  in stage IV, and to even lower values in stage V. Thus, in order to design a useful model to predict vacancy concentrations even at very high strains, it would be desirable to extend Saada's model by terms of dislocation and vacancy annihilation, and let the coefficient  $\alpha_i$  constant in the interest of a sound physical concept. This would be especially important within actual research on severe plastic deformation techniques where mechanisms of vacancy formation seem to play an eminent role for the realization of ultrafine grained structure with outstanding mechanical properties (see also section 4) [18].

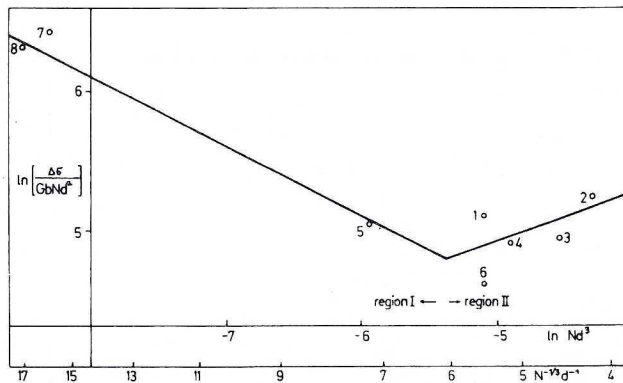


Fig. 5. Hardening in metals due to the growth of vacancy loops during coarsening process. While at first the strength decreases due to Orowan mechanism (left side), the strength starts to increase when a critical distance between loops gets smaller than  $N^{-1/3} \times d^{-1} = 6$  (Kirchner, [21])

## 2. Direct effects of vacancies (-agglomerates) to strength(ening)

As long as the vacancies are produced as single ones, their effect on macroscopic strength is not too large. This arises from the fact that the single vacancy defect only spans a few atomic distances and can be overcome by thermal activation of the moving dislocation. Accordingly, only small hardening effects have been observed experimentally in fast quenched samples [19]. As a contrast, agglomerates of vacancies imply long range elastic strain fields which give rise to athermal "Orowan" type interaction as it has been modeled for isotropic (Krupa [20]) and anisotropic

(Kirchner [21]) lattices. In isotropic lattices (i.e. cubic ones) there is a high number of crystallographically dense packed planes, and both the formation of vacancy agglomerates and interaction with moving dislocations will occur in all of these planes. In anisotropic lattices (e.g. hcp ones) only a few dense packed planes exist where the vacancies can be formed as planar loops, and where the dislocations can move. This will lead a strong concentration of vacancy-dislocation interaction in these planes which makes vacancy hardening effect up to 10 times larger in anisotropic (e.g. hcp) lattices than in isotropic (cubic) ones (compare Fig. 6 from [25]). Apart from different exponents  $a$  representing the isotropic ( $a = 2/3$ , [22]) and the anisotropic ( $a = 1/2$ , [21]) case, all models [20—22] arrive at the same relation for loop hardening from Orowan type interaction as

$$\Delta\tau = \mu b/k \quad N^a \quad d^{(3a-1)}, \quad (2)$$

where  $N$ ,  $d$  mean the density and the diameter of loops, respectively. This equation is valid for hardening from loops during growth and/or coarsening. For coarsening solely (i.e. constant total loop area  $f = N d^2$ ), equation (2) reduces to

$$\Delta\tau = \mu b/k \quad f^a \quad d^{(a-1)}. \quad (3)$$

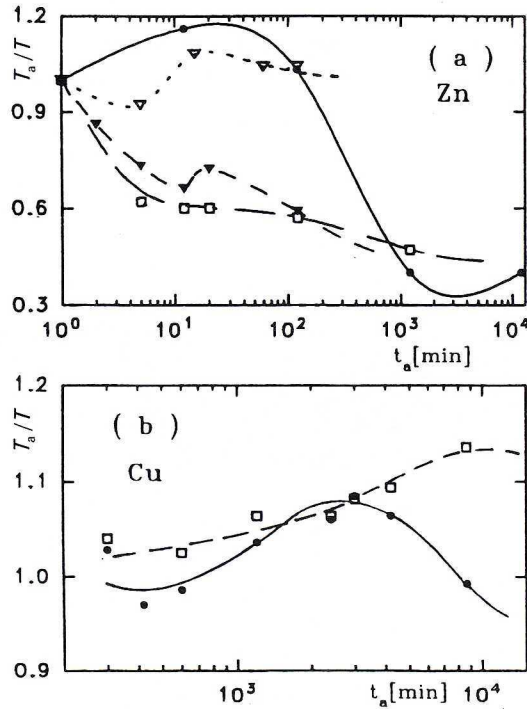


Fig. 6. Isotherms of hardening at  $T_a = 293$  K after pre-deformation at  $T_d$  by shear strain  $\gamma$ , and after quenching, in single crystals of Zn and Cu (from [25]). a) Zn:  $T_d = 77$  K  $\gamma = 0.1$ ,  $\gamma = 0.5$ ,  $\gamma = 1.2$ , and — quenched. b) Cu:  $T_d = 77$  K  $\gamma = 0.1$ ;  $T_d = 293$  K,  $\gamma = 0.1$

According to these equations, hardening is only expected in case of growth of loops since coarsening yields always a softening effects. However, if the mean loop distance shrinks to less than 10 loop diameters, the strain fields of coplanar loops overlap which leads, by a change of exponent  $a$  and coefficient  $k$ , to a hardening effect even in case of coarsening which has been stresses by K i r c h n e r [21] for the anisotropic case,  $a = 4/3$ . Fig. 5 shows the effects predicted by K i r c h n e r's theory together with anneal hardening experiments in quenched Mg for the case of widely (left side) and closely distanced coplanar loops during coarsening process. The group of the authors proved the same effect to occur for the case of deformation induced vacancies by numerous experiments on different metals for both poly- and single crystals [23—25]. Fig. 6 shows the occurrence of anneal hardening by examples of single crystals of Zn and Cu deformed by different extents at different deformation temperatures. Comparing the results of Zn and Cu, a marked shift in the annealing time to reach the maximum can be recognized which could be quantitatively ascribed to the different self diffusion conditions in these metals [25]. As already expected by K i r c h n e r [21], many of the curves show a final softening effect where pure coarsening conditions are no longer true because of successive annihilation and thus depletion of vacancies occurring at the large annealing times. Another prove of this concept is the fact that the observed hardening effect decreases when the predeformation done before annealing is increased; it seems that the higher the dislocation number produced during predeformation the easier the free vacancies will be trapped by the dislocations and finally annihilated there. Thus these

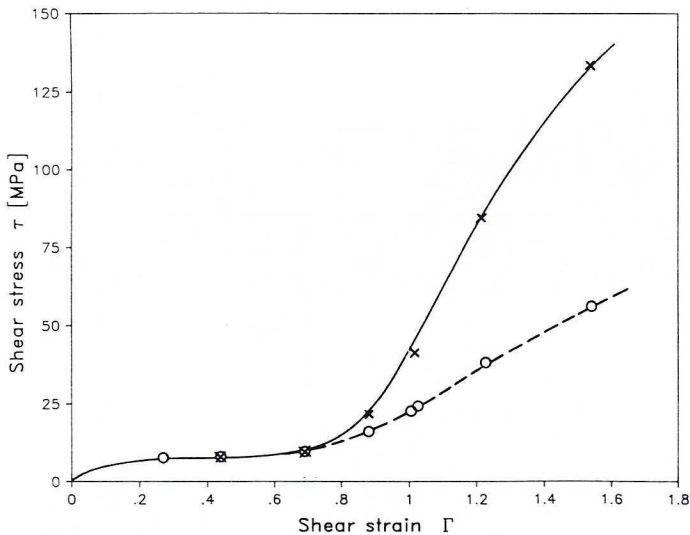


Fig. 7. Hardening curves in single slip oriented Zn single crystals. Full line: Experimental results from compression test. Dashed line: Hardening concluded from calorimetric measurements of dislocation density done at ahear strains of  $\gamma = 0.28, 0.44, 0.68, 0.88, 1.02, 1.05, 1.22,$  and  $1.54$  (see text and [11])



vacancies would no longer be able to agglomerate and to hinder the dislocation movement. Similar, although smaller effects have been observed in pure Al [26].

There are clear indications that the generation of vacancy agglomerates does occur already during deformation and that these are responsible for a considerable contribution of overall hardening. This certainly follows from atom probe experiments by Perturbed Angular Correlation (PAC, Collins [4]) in as-deformed Au and Al which shows a clear peak at the temperature of multivacancy annealing. It also follows from calorimetric studies of Zehetbauer et al. [11] in low temperature deformed Zn single crystals where the stored energies from annealing of double vacancies ( $E_{\text{stor}}(\text{vac})$ ) and dislocations ( $E_{\text{stor}}(\text{dis})$ ) have been measured. According to Seeger & Kronmüller [27] the ratio  $E_{\text{stor}}(\text{dis})/E_{\text{expend}}$  ( $E_{\text{expend}}$  is the total energy expended during plastic deformation) rules the hardening coefficient which, however, results as about a factor 2 too small compared to the hardening being directly observed (Fig. 7). This behaviour suggests that also the vacancies (-agglomerates) have to be considered for the stored energy being responsible for hardening, which means that these must have been generated already during deformation.

### 3. Indirect effects of vacancies (-agglomerates) to strength(ening)

Returning to Fig. 2a, it can be clearly seen that a large number of vacancies is produced during cold working of metals especially when strain gets very large. Moreover, it seems that the concentration of all these vacancies (-agglomerates) stops to increase as far as stage  $V$  of deformation is reached. Since the inspection of TEM images reveals small angle boundaries to have formed, it can be said that climb of dislocations is initiated when both a critical stress as well as a critical vacancy concentration has been exceeded due to plastic deformation. Then it is plausible that both quantities, i.e. the vacancy concentration and the dislocation density will level out at the highest strains achieved.

The important role of indirect effects of deformation vacancies being present is also reflected in work hardening models describing large strain strengthening behaviour. Because of increasing heterogenization of dislocation structure during deformation, edge dislocations will be concentrated in cell walls with certainly higher dislocation density that it is in the cell wall (Fig. 8). Thus the vacancy agglomerates are predestined to be generated in cell walls.

One of these models [29] explicitly accounts for the vacancy controlled climb of dislocations in describing stage  $V$  of deformation. It is shown that the climb mechanisms at ambient deformation temperatures and higher (up to  $0.5 T_m$ ) will operate only when the actual vacancy concentration generated by deformation will be drastically higher than that of thermal equilibrium (Fig. 9, measuring points); otherwise, the work hardening behaviour being typical of stage  $V$  can never be reflected (Fig. 9, full lines). The values of vacancy concentration reached in steady state of stage  $V$  at different deformation temperatures are collectively presented in

Fig. 10, and are compared with the respective thermal vacancy concentration. It is clearly seen that the vacancy concentration is several orders of magnitude higher than that in thermal equilibrium as long as the deformation temperature is lower

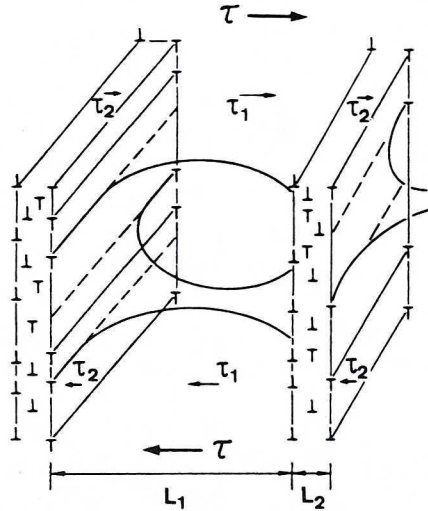


Fig. 8. 3-dimensional cell structure with cell walls and cell interior, and moving dislocations (after Nixon et al. [28])

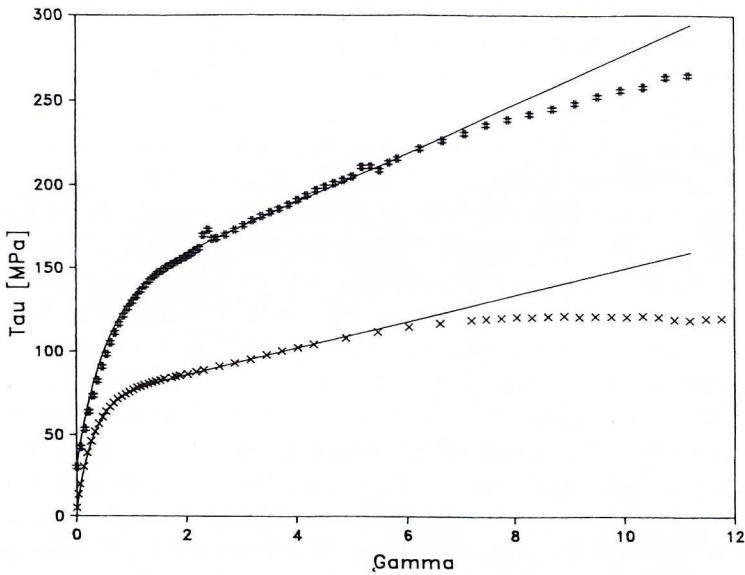


Fig. 9. Hardening of Cu in plastic deformation up to large strains. If one does not consider the high number of deformation induced vacancies in modelling of large strain strengthening, the experimental data (# for 77 K, x for 293 K) cannot be reflected at all (from [29])

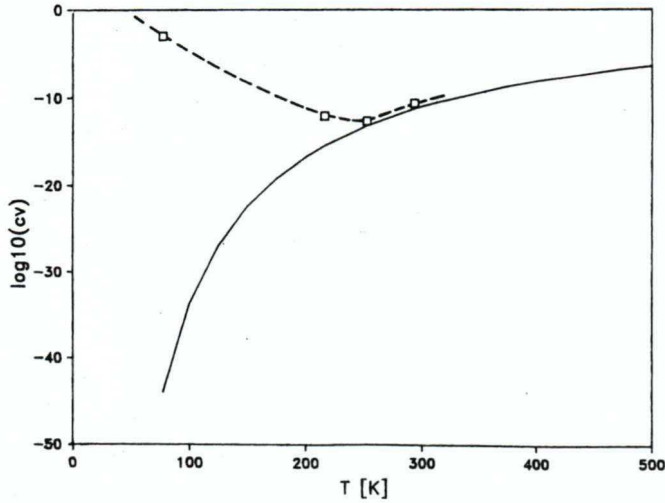


Fig. 10. Vacancy concentration from deformation up to saturation for different deformation temperatures (experimental & theoretical data, dashed line) as compared to the thermal vacancy equilibrium concentrations (calculated values, full line), for pure Al. Note that the dashed line follows the full one for deformation temperatures  $T \geq 0.28 T_m$ . (from [29])

than  $0.28 T_m$ . With increasing temperature, however, the intrinsic vacancy concentration more and more approaches the value of thermal equilibrium which seems quite sensible having in mind the marked onset of diffusion at that temperature due to mobilization of single vacancies.

When the deformation will not be carried out in a continuous way, i.e. performed by single passes like in rolling, extrusion and wire drawing, the presence of supersaturated deformation vacancies may allow for additional dislocation climb and -annealing during the unloading between the deformation passes. Then, with the next deformation pass, the original dislocation density and/or long range internal stress cannot be reached because of the limited extent of the deformation pass. This has been shown by experiments in Cu and Al having studied the influence of the magnitude of rolling pass to the resulting work hardening characteristics [30]. The smaller the deformation pass chosen, the more static recovery processes are probable to occur during unloading leading to a deterioration of strength [30], internal stress or even dislocation density [31].

#### 4. Practical importance of large strain deformation — research: nanocrystalline materials

The evolution of microstructure during large strain deformation is governed by the successive formation of tilt walls from original cell walls so that misoriented lattice areas (“cell block”) develop which shrink in size with proceeding deformation [32]. Large strain work hardening (stage IV) then arises from the continuous shrink

of mean free path of dislocations which cannot pass tilt walls with a misorientation  $\geq 10^\circ$  [32], although other sources for hardening may exist (see below). The deformation induced vacancies will become effective in stage V where they launch climb of edge dislocations in tilt walls and thus the formation of real subgrain walls with reduced internal stresses. While at conventional large strain techniques such as rolling, compression and torsion, the final cell block shape is not equiaxed and not smaller than 200 nm in the short axis [32], the situation changes when more complex deformation techniques (“Severe Plastic Deformation- SPD”) will be applied (Fig. 11). From these, the methods of High Pressure Torsion (HPT) [18], Equal Channel (ECA) pressing [18], and Cyclic Extrusion Compression (CEC) [33] have been mostly applied up to now. By special design of deformation die restricting free flow of material, all these SPD techniques more or less provide the presence of hydrostatic pressure component (HPC). The higher the PHC, the more it prohibits the material from failure, allowing for ultrahigh deformation and formation of ultrafine-grained or nanocrystalline structure. It is then possible to produce massive samples with microstructures with high angle equi-axed subgrains down to 20 nm size (Fig. 13) which will be therefore attractive for industrial application [18], not at least due to their final strength of samples being in pure metals by about 20%, in alloys up to a factor 3 higher than in conventional large strain cold working. The number of supersaturated vacancies from plastic deformation seems to be extraordinarily high in materials produced by these techniques. The density of deformation induced defects as measured by electrical residual resistivity (which is sensitive to both deformation induced vacancies as well as dislocations [34]) exceed that from X-ray Bragg Profile Analysis (XPA) (which is exclusively sensitive to dislocations [35]) by a factor of 1.7 in cold rolled Cu, but by a factor of 2.5 in ECA pressed Cu [36]. Moreover, since the XPA signal indicates the highest dislocation density to be the same in both deformation modes ( $\rho \approx 1.7\text{--}1.8 \times 10^{15} \text{ m}^{-2}$ , table 1 [37]), it seems that the vacancy concentration in ECA pressed Cu is markedly higher than that found in cold rolled Cu. In view of the break-down of Hall-Petch relation repeatedly observed in nanostructured materials with grain sizes smaller than 50 nm [38], these results suggest that the increase of macroscopic strength in SPD materials arises, at least partially, from direct interaction of deformation induced vacancies and/or vacancy agglomerates with dislocations. The reason for the enhanced density of deformation induced defects, namely vacancies, is thought to be the presence of an enhanced hydrostatic pressure since it decreases the mean lattice distance and thus the ability of vacancies to migrate through the lattice. Annihilation of both vacancies as well as of dislocations would be therefore restricted. The observed levelling out of dislocation density does not fit to this picture, but this could be a measuring problem due to the limits of the XPA methods for vacancy agglomerates and/or dislocation loops smaller than 5 nm [35]. On the other hand, due to recent measurements of microhardness in HPT and ECA deformed Cu [39, 40] exhibiting a levelling out of (although enhanced) strength up to ultrahigh strains, it cannot be excluded that processes of static recovery occur during the breaks in between single deformation

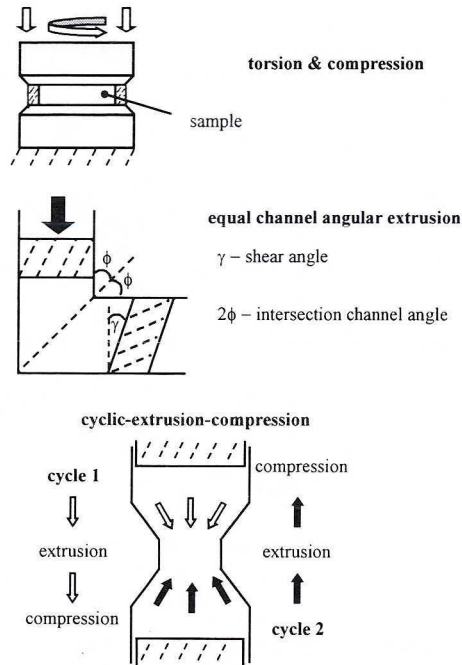


Fig. 11. Different techniques of severe plastic deformation providing ultrafine grains up to nanometer scale

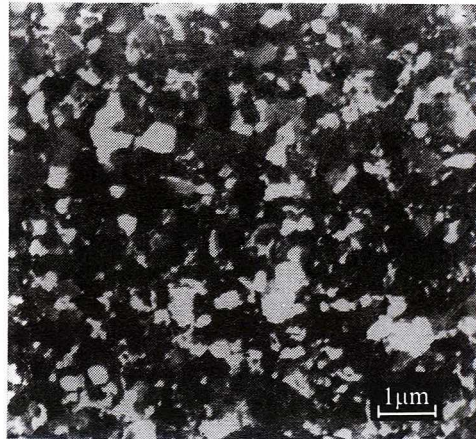


Fig. 12. Subgrains of size 200–300 nm with high angle boundaries, in ECA pressed Cu by  $\epsilon = 12$  (from Mingler et al. [41])

passes, allowing for additional annihilation of dislocations, i.e. levelling out of dislocation density, under pressure free condition. The situation could be analogous to that described above for iterative way of conventional large strain deformation. Further experiments have to be carried out in order to finally decide on this question.

TABLE

Dislocation density and grain size as a function of plastic strain, for conventional large strain deformation (rolling) and severe plastic deformation (equal channel angular pressing) in polycrystalline Cu 99.99%

Deformation method, True strain $\epsilon$	Dislocation Density (by XPA [37])	Grain size by TEM [41, 42])
<b>ROLLING</b> $\epsilon = 0.79$ $\epsilon = 2.45$	$1.2 \times 10^{15} \text{ m}^{-2}$ $1.8 \times 10^{15} \text{ m}^{-2}$	820 nm 590 nm
<b>ECA-PRESSING</b> $\epsilon = 2$ $\epsilon = 12$	$1.7 \times 10^{15} \text{ m}^{-2}$ $1.7 \times 10^{15} \text{ m}^{-2}$	300 nm 275 nm

### 5. Summary & conclusions

The main aspects of the present paper may be summarized as follows:

1. The density evolution of deformation induced vacancies is described best by the model of Saada. At higher strains, however, it does not account for thermally activated annihilation processes of vacancies and dislocations being necessary for a correct prediction of their concentration with increasing plastic strain.

2. There clearly exist direct effects of deformation induced vacancies to the macroscopic strength. While there is only small influence of single vacancies, that of vacancy agglomerates can reach up to a factor 2—3 (e.g. in alloys and/or in single crystals).

3. Stage *V* of deformation is characterized by the consumption of deformation induced vacancies through climb processes of edge dislocations. This enables the dislocations to annihilate, too. In iterative deformation modes, these effects will be enhanced due to unloading in between the deformation passes.

4. During severe plastic deformation (large strain cold working under elevated hydrostatic pressure) very high concentrations of deformation induced vacancies seem to be reached. Because below a grain size of 50 nm, the Hall-Petch relation appears no longer to be fulfilled, the high number of vacancies i.e. particularly vacancy agglomerates could be responsible for the enhanced strength in SPD nanocrystalline materials. This idea is confirmed by quantitative comparison of the strength increase observed in SPD with that from direct interaction of vacancy agglomerates with dislocations: both amount to about 20% in pure metals, but reach about a factor 3 in alloys.

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