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Deformation Mode Change in D-T Neutron Irradiated Ni and Au⁺

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Abstract

The process of the deformation structure development in D-T neutron irradiated Ni and Au was investigated. In Ni irradiated to fluences ranging from 1×10^{21} to 10×10^{21} n/m², and in Au from 0.5×10^{21} to 5×10^{21} n/m², the transition in the deformation process from dislocation channeling to that by cell formation was observed with an increase in the deformation amount. This transition took place with larger deformation in specimens irradiated to higher fluences. In the specimens for a fluence lower than the minimum fluence of these ranges, the deformation by cell formation was observed to be continued until the specimen broke, whereas the deformation by dislocation channeling for a fluence higher than the maximum fluence, respectively. The deformation mode transition is illustrated in a diagram as a function of both irradiation fluence and deformation amount.

The difference in the deformation structures can be understood to have resulted from the difference in the stacking fault energy. The shape of the stress-strain curves and material ductility were also discussed correlating the deformation structures.

1. Introduction

Application of the miniaturized mechanical test technique^{1),2)} is highly practical for the investigation of the correlation between the mechanical property changes and deformation structure changes, because it is possible to observe the microstructure of the test specimen after applying a designed amount of deformation. The present authors have reported the correlation between the deformation structure and mechanical property change for various metals³⁾⁻⁵⁾.

Neutron irradiation produces a large variety of deformation structures in metals^{6),7)}.

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The defect structure varies widely from metal to metal and irradiation conditions, even with irradiation of the same fluence. The different defect structures will result in different mechanical property changes.

In order to investigate the correlation between the deformation structures and mechanical property change, tensile tests and observation of deformation structure were carried out on Ni and Au using miniaturized tensile specimens.

2. Experimental Procedures

2.1 Specimens

Miniaturized tensile specimens were prepared from Ni of 99.99% purity and Au of 99.999% purity. These specimens were prepared by punching from cold rolled sheets of 0.2 mm thickness. After punching, Ni and Au were annealed at 1170 K and 770 K for 1.8 ks in vacuum, respectively. The dimensions of the miniaturized specimen were 12.5 mm in length and 2.3 mm in width with gauge section 1.2 mm \times 5.5 mm.

2.2 D-T neutron irradiation

Specimens were irradiated with D-T neutrons from a Rotating Target Neutron Source (RTNS-II) at Lawrence Livermore National Laboratory. The irradiation was carried out up to 4×10^{22} n/m² at 300 K by arranging specimens systematically at locations with various neutron flux density in the irradiation capsule. The ratio of the highest to the lowest neutron flux density in the capsule was larger than 2 orders.

2.3 Tensile tests and structure observations

Tensile tests were performed at 300 K, at a strain rate of 5×10^{-4} s⁻¹. The deformation structure observations were conducted on these specimens using an optical and electron microscopes after applying designed amount of elongation, 2 and 5%, and up to the limit of the uniform elongation. The TEM (transmission electron microscopy) specimen was prepared from the parallel portion (1.2 mm wide) of the tensile specimen by electrolytical thinning. In addition to both optical and electron microscopic observations at a given deformation amount, the development of the slip pattern on an identical area of the specimen surface was successively observed by stretching the specimen with a micrometer-head under an optical microscope.

3. Experimental Results and Discussion

3.1 The variation of the stress-strain curves of Ni and Au irradiated with D-T neutrons

In Fig. 1, sets of the stress-strain curves of specimens irradiated with D-T neutrons for several fluence levels are shown for Ni and Au, respectively.

(1) Nickel

As seen in Fig. 1(a), with increase in the fluence the yield stress increased and the uniform elongation limit decreased to almost a half of the unirradiated specimen. These curves show that the stress levels where the uniform elongation terminates are almost the same for all of the irradiated specimens. This result suggests that the ultimate tensile strength of these specimens is limited to that of unirradiated specimens. As far as the yield stress increased by the irradiation is lower than the work hardening limit in tensile deformation, the ultimate strength may depend on the uniform elongation limit which is controlled by the work hardenability. In this case, the stress strain curves of the irradiated specimens can be regarded as that of an unirradiated specimen whose first half is cut off at a stress level corresponding to the yield stress of the irradiated specimen and shifted to the left. This is equivalent to reloading the annealed specimen after applying a certain amount of deformation.

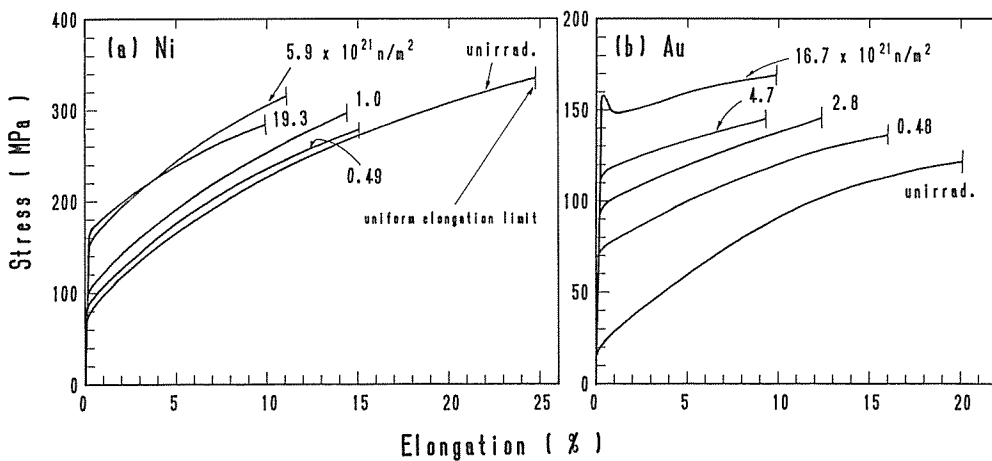


Fig. 1 Stress-strain curves for D-T neutron irradiated Ni and Au.

(2) Gold

For cases of relatively lower fluence, the changes of the stress-strain curves of Au are almost the same as that of Ni as seen in Fig. 1(b). For higher fluence, in addition to the increase in the yield stress and the shift of the curves to the left, their slope, viz., the strain hardening, becomes much slower than in Ni where the yield drop appeared. For the highest fluence in the present experiment, the yield stress exceeds the tensile strength (in a true stress measure) of the unirradiated specimen and uniform elongation limit showed a slight increase in contrast to the expectation. In general, the uniform elongation limit can be roughly estimated from the slope of the stress-strain curve, i. e., if the flow stress σ is presented by $\sigma = k (\text{strain})^n$, where k is a constant, the uniform elongation limit is equal to n .

Thus, the smaller elongation limit should be expected from smaller work hardening exponent. The mismatch with this expectation in Au is the most marked difference in the curves between Au and Ni, and is discussed in the later section.

3.2 Defect structured of irradiated specimens

(1) Nickel

Very small defect clusters of 0.5 to 3 nm can be seen as in Fig. 2. These small clusters are classified into two types, clusters larger than 2 nm are the interstitial type dislocation loops and other smaller clusters are stacking fault tetrahedra (SFT). The number of these clusters increased linearly with neutron fluence, and their number density was $3 \times 10^{22}/\text{m}^3$ at $1 \times 10^{21} \text{ n/m}^2$.

(2) Gold

In irradiated specimens, SFT of a mean size of 1.5 nm and a smaller number of large interstitial type dislocation loops approximately 4 to 8 nm in diameter are observed as shown in Fig. 3. The number of these defect clusters also is linearly proportional to the neutron fluence as the same in Ni, and their number density was $2 \times 10^{22}/\text{m}^3$ at $1 \times 10^{21} \text{ n/m}^2$.

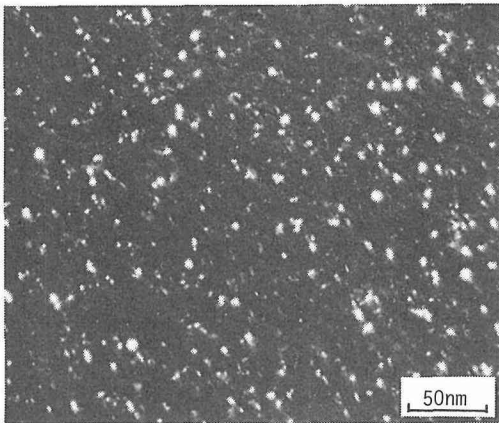


Fig. 2 Defect structures of Ni irradiated with D-T neutrons up to $3.8 \times 10^{22} \text{ n/m}^2$

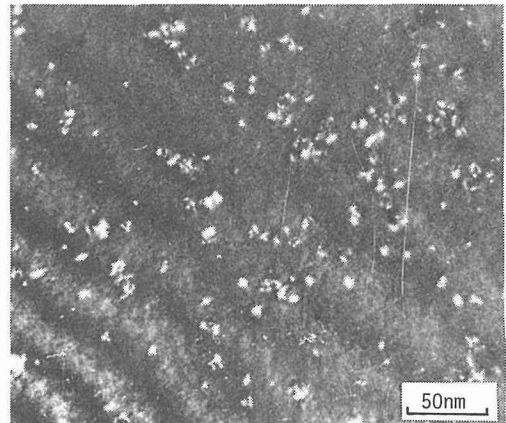


Fig. 3 Defect structures of Au irradiated with D-T neutrons up to $4 \times 10^{21} \text{ n/m}^2$.

3.3 Variation of slightly deformed structure by fluence

The deformation patterns on the surface and the dislocation structures of Ni and Au were observed under an optical and electron microscope, respectively, after subjecting to a slight tensile deformation (2%).

(1) Nickel

The surface slip patterns and dislocation structures are shown in Fig. 4 and 5. In the non-irradiated specimens, a well developed cell structure could be observed by electron microscopy without showing any detectable slip traces on its surface. No remarkable slip traces remained observable for irradiation up to $5 \times 10^{20} \text{ n/m}^2$, however, during this stage the cell

size became smaller and dislocations tended to be arranged along a straight line parallel to the slip plane with the increase in the fluence. The formation of the dislocation arrays parallel to slip planes continued at higher fluence, and widely spaced slip bands became observable at a fluence about 10^{21} n/m². For the specimens irradiated higher than 10^{22} n/m², slip patterns became more distinct with many crossings, and their corresponding dislocation structure was composed of well developed dislocation channels.

A very clear transition of deformation mode from the homogeneous slip forming cellular structure to the highly localized slip to form dislocation channeling was observed.

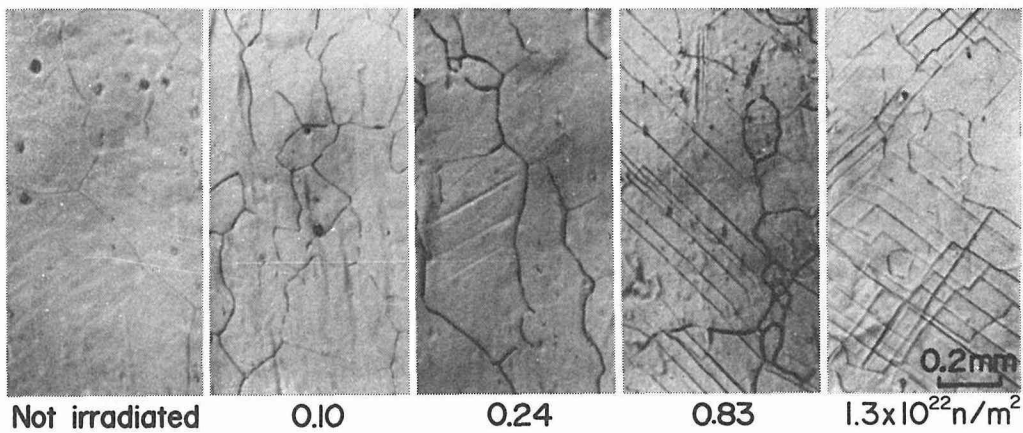


Fig. 4 Slip patterns of slightly deformed Ni.

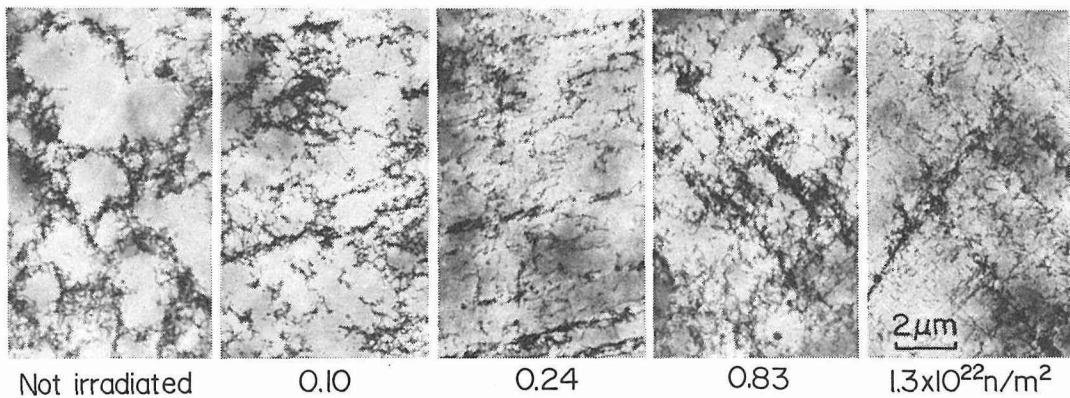


Fig. 5 Deformation dislocation structures of Ni.

(2) Gold

As seen in Fig. 6 and 7, the surface of the nonirradiated Au after deformation of 2% was covered with uniformly distributed fine slip lines. These slip lines tended to conglomerate and formed clusters with the increase in the fluence up to $0.3 \times 10^{21} \text{ n/m}^2$. For the fluence higher than this level, zig-zag slip lines developed with spacing of a few μm between each other. The dislocation cell size decreased from $1 \mu\text{m}$ of nonirradiated specimen to $0.5 \mu\text{m}$ of that irradiated with $0.5 \times 10^{21} \text{ n/m}^2$. In the specimens irradiated by higher fluence ($1.8 \times 10^{21} \text{ n/m}^2$), whose surface was covered with zig-zag slip lines, the dislocation channels were clearly

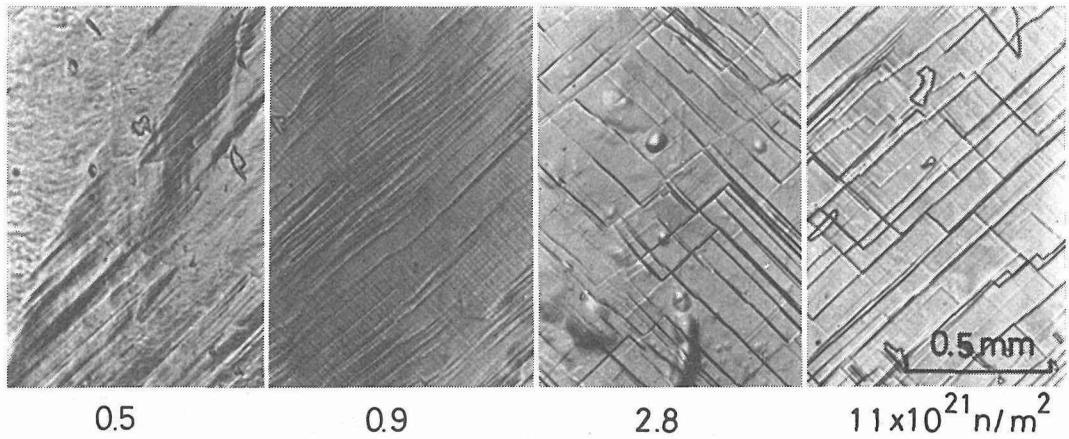


Fig. 6 Slip patterns of slightly deformed Au.

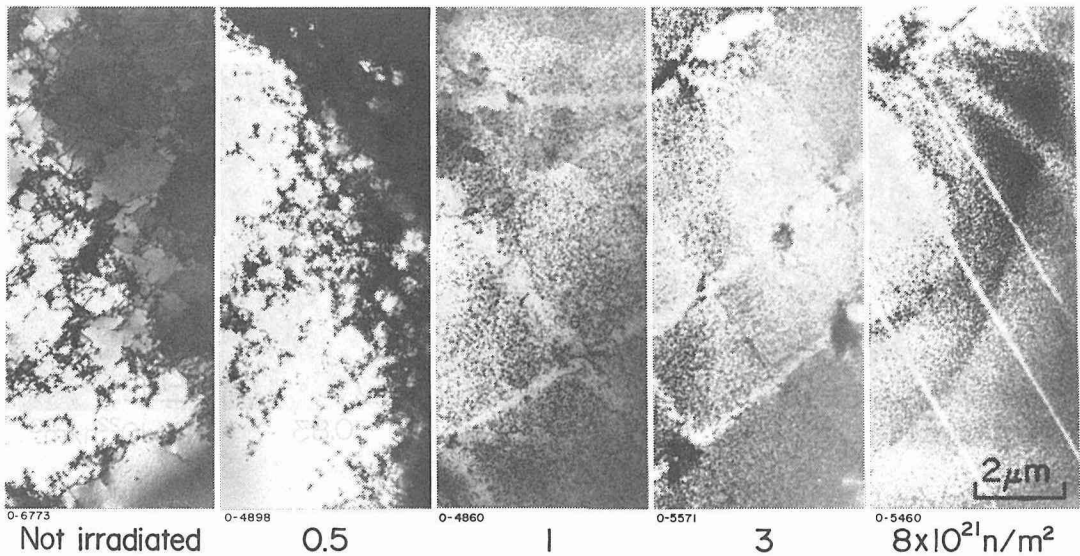


Fig. 7 Deformation induced location structures of Au.

observed and dislocations were seen only in the intersection of the channels. Except for these areas in the channel, almost no dislocations and no defect clusters (SFT) could be observed. No change in the irradiation induced defect structure could be observed outside of the channels.

The deformation structures of the clear dislocation channels in Au renders a contrast to the structures containing dense dislocation tangles in the channels and matrix in Ni. This corresponds to the smaller work hardening during the channeling deformation in Au.

3.4 Deformation structures at moderate and large deformation amounts

The identical area of the surface was successively observed under an optical microscope for Au irradiated up to $1.1 \times 10^{22} \text{ n/m}^2$. In the early stages of the deformation, for elongation less than several percents, the spacing of slip lines rapidly decreased as shown in Fig. 8. However, for larger deformation, their contrast, width and spacing almost remained unchanged until the specimen broke. The optical and electron microscopic observations suggest that there are two deformation modes. One is the usual deformation process in which the uniform slip takes place to form cell structure. Another one is the dislocation channeling deformation in which the development of widely spaced slip lines is observed under an optical microscope as mentioned above. In the specimen irradiated to a lower fluence, only the usual deformation mode can be observed until the specimen breaks. Whereas in the specimens irradiated to a higher fluence, the deformation is first preceded by the dislocation channeling and at larger deformations this deformation mode changes to the cell formation. The deformation amount where this transition takes place depends on the degree of the irradiation hardening. The higher the irradiation fluence is, the larger the deformation amount for the transition becomes. The same deformation mode change as seen in Au was observed in Ni, however the development of the slip pattern is not obvious in the specimens of lower fluence.

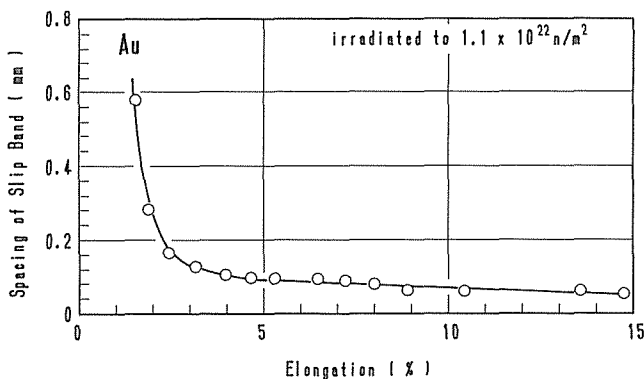


Fig. 8 Variation of channel spacing as a function of the deformation amount.

3.5 A diagram for the deformation mode transition

The observation for the deformation structure change in Au and Ni were conducted at various amounts of deformation, viz., at 2 and 5%, and at the uniform elongation limit (around 10%) for specimens irradiated to various fluence levels. The structures developed at the uniform elongation limit were observed for the uniformly elongated part of the specimen.

The types of the deformation mode for fluence and deformation amounts are depicted as shown in Fig. 9(a) and (b). These diagrams show that the deformation mode transition from the dislocation channeling to the usual slip with the formation of the dislocation cell structure taking place before the fracture for the neutron fluence ranging from 0.5×10^{21} to 5×10^{21} n/m² in Au, and from 1×10^{21} to 10×10^{21} n/m² in Ni.

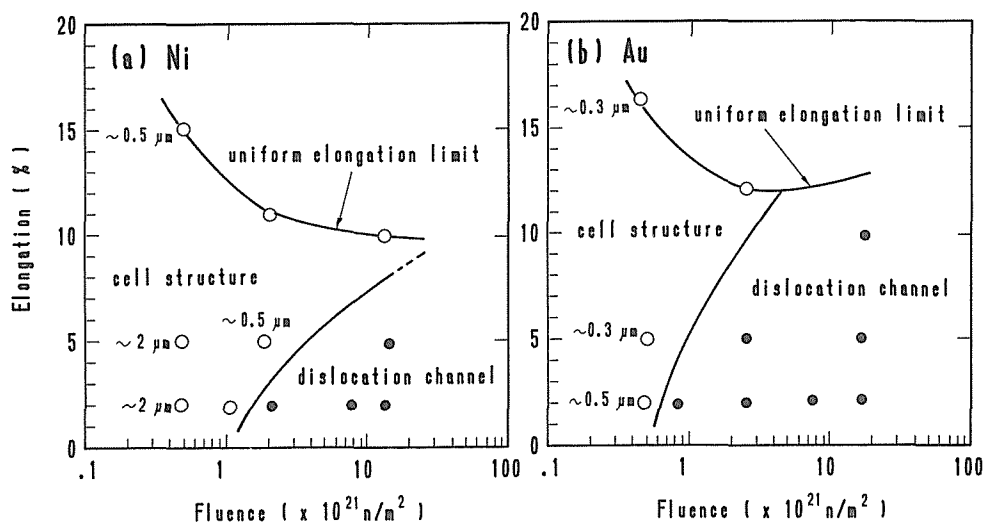


Fig. 9 Deformation mode transition diagram for Ni and Au.

3.6 Deformation mechanism of irradiated Ni and Au

The general feature of the stress-strain curves for irradiated metals relating to the deformation mode change, can be discussed by using the scheme shown in Fig. 10. Let the fracture stress level of the nonirradiated metal be σ_{\max} (viz., it corresponds to the limit of work hardening). If the yield stress of the irradiated metal is smaller than σ_{\max} , the flow stress can increase up to σ_{\max} with an increase in the deformation amount through the work hardening process. This work hardening process starts after the channeling deformation finishes and this process may be somewhat different from that of unirradiated metals due to the existence of the dislocation channels already formed by the leading process and the defect clusters. Though the initial condition is different by irradiation fluence, the shape of the second half of the stress-strain curve after yielding does not change, if the work necessary to bring about the plastic deformation depends on the flow stress. The experimental results

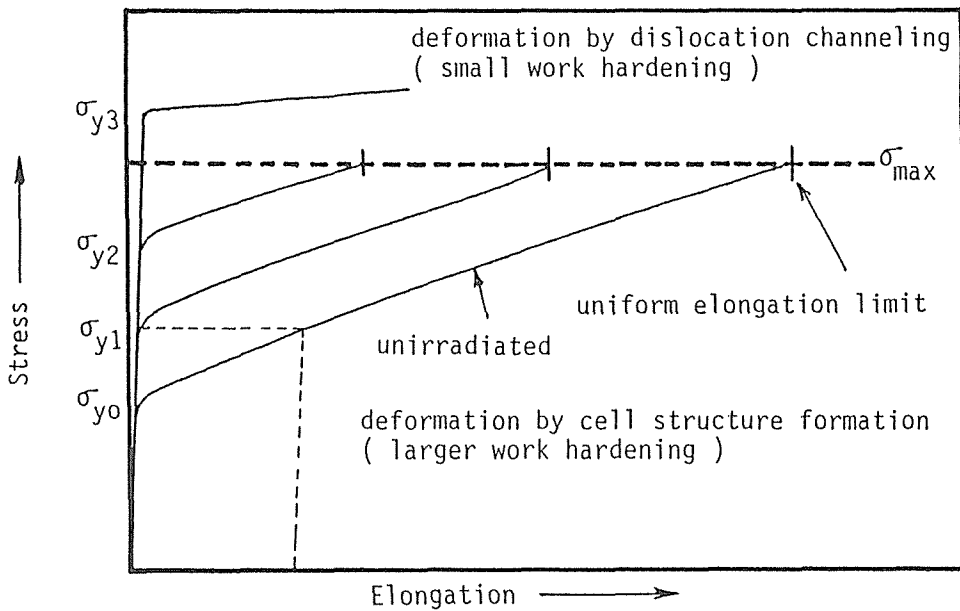


Fig. 10 Scheme for deformation mode change in neutron irradiated metal.

well corresponded to this prediction as mentioned before.

On the other hand, when the highly irradiated specimens, whose yield stress is larger than σ_{max} , are deformed in tension, they cannot be work hardened additionally by the tensile deformation, i. e., by forming dislocation cells, because their whole volume is already much more hardened by the irradiation. In these specimens, the dislocations can glide in the channel, which is formed by large stress concentration sufficient to eliminate the defect clusters.

After a certain number of dislocations slips, a larger stress required to move dislocations against the back stress of the dislocation pile-ups at grain boundaries, and the formation of another new channel must be necessary. In this slip process, until the crossing among dislocation channels take place the flow stress increment may be very small, because the dislocations can slip without cutting each other in the channel. The transposing of the dislocations from the channel is difficult in low stacking fault energy metals such as Au, however it may be easy in metals of high stacking fault energy metals such as Ni. The difference in the readiness of the transposing of the dislocations from the channels corresponds to the work hardening rate and the deformation dislocation structures. In low stacking fault energy metals such as Au, because the cross slip is difficult to take place, the interaction among dislocations on parallel slip planes in a channel may be smaller. This agrees with the observations in which few dislocation tangles remained in the channels except for the intersections of the channels in Au, as is seen in Fig. 5.

Therefore, the deformation mode change, from the usual slip deformation to the dislo-

cation channel deformation, suggests that the conventional criterion for the ductility, i. e., the uniform elongation limit is estimated from the work hardening exponent, becomes no more valid for channeling deformation in highly irradiated metals.

4. Conclusion

The deformation structure change in the D-T neutron irradiated Ni and Au deformed at 300 K was investigated under an electron microscope by using miniaturized tensile specimens after applying a designated amount of deformation and successive observations under an optical microscope. The results obtained were summarized as follows.

- (1) The transition from the deformation process by the dislocation channeling to that by cell structure formation was observed in the specimens irradiated to the fluences ranging from 1×10^{21} to 10×10^{21} n/m² in Ni and 0.5×10^{21} to 5×10^{21} n/m² in Au.
- (2) In the specimens irradiated to the fluences higher and lower than these levels, the deformation by the formation of the dislocation channels and dislocation cells, respectively, were observed to continue until the specimen broke.
- (3) Deformation mode change was illustrated in a diagram as a function of deformation amount and the irradiation fluence.
- (4) In the early stages of the development of the slip patterns, their spacing rapidly decrease, and after deformed to several percents they decrease very slow. In Au, spacing was saturated at about 60 μ m in the uniformly elongated part of specimen, and at about 40 μ m in the fractured portion.
- (5) The difference in the detailed deformation structures especially in the dislocation channel between Ni and Au, can be understood as a result from the difference in the stacking fault energy.

Acknowledgments

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