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ДУБНА

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**233 MeV Ne ION IRRADIATION EFFECTS  
ON THE MECHANICAL PROPERTIES  
OF COPPER**

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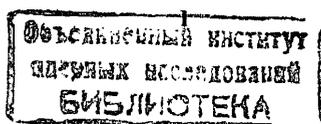
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## 1. Introduction

The defect structure of copper irradiated by neutrons or charged particles has been examined extensively for many years. The interest in this material is explained by that it may serve as a model material for the investigation of the defect structure in irradiated fcc metals, and by its possible use in fusion reactor technology in the near future. The defect structure of irradiated copper has been investigated mainly by electrical resistivity measurements [1-5], transmission electron microscopy [6,7] and by the measurements of mechanical properties [8-10]. Recently a growing interest has been shown in heavy-ion irradiation experiments [11-14]. The results extensively reveal the advantages and limitations of heavy-ion irradiation in the simulation of neutron irradiations, although a lot of details have not been so far clarified. The investigation of the mechanical properties of materials irradiated by energetic heavy ions is also in progress at the Joint Institute for Nuclear Research (Dubna). This program is based on the U-400 cyclotron. The irradiation system developed makes it possible to perform ion bombardments under controlled conditions [15], and our first results for Ne ion irradiated nickel [16] have shown the possibilities of this project. In the present work the deformation behaviour of polycrystalline copper irradiated by energetic Ne ions is investigated. The main experimental results are presented here, and a more detailed analysis will be made in the following paper.



## 2. Experimental

The irradiation facility and the experimental procedure have been described elsewhere [15-17]. Therefore only the most important things will be given here. Tensile specimens were prepared from a 30  $\mu\text{m}$  cold-rolled sheet. The material used was 99.998 wt% purity copper, containing Fe(0.0013 wt%) and Ni(0.0004 wt%) as main impurities. The specimens were of standard form for tensile test, with a 15 mm gauge length and 3 mm width. Before irradiation these were annealed at 350  $^{\circ}\text{C}$  for 1h in vacuo. This temperature produced an entirely recrystallized grain structure with a mean intercept grain diameter of 5  $\mu\text{m}$ . The irradiations were performed in air by the accelerator Ne ion beam passing through a thin aluminium window. The ion energy at the entry surface of the specimens was 233 MeV. During the ion bombardment the specimens were cooled by a room temperature air jet, so that the temperature did not exceed 100  $^{\circ}\text{C}$ . The temperature was indirectly controlled on special samples by measuring the changes in length when the ion beam was switched on. The depth distribution of the defect production cross-section for a Ne projectile and Cu target is shown in fig.1. This distribution function was calculated by a computer code [18] using the relation

$$\sigma_d = \sum_{i,j} \Phi(E_i, \theta_j, x) \int_{E_d}^{\gamma E_i} \frac{d\sigma(E_i, \theta_j, T)}{dT} \gamma(T) dT, \quad (1)$$

where  $\sigma_d(x)$  is the Frenkel-pair production cross-section per atom, as a function of the projectile range;  $\gamma(T)$  is the damage function, that is the number of Frenkel-pairs produced by a primary knock-on host atom of energy  $T$ ;  $d\sigma/dT$  is the differential cross-section for producing host atom recoil of energy  $T$ ;  $E_d$  is the effective threshold energy for a displacement;  $\gamma E_i$  is the maximum energy transferred; and  $\Phi(E_i, \theta_j, x)$  is the distribution of the projectile as a function of energy  $E_i$  and angle  $\theta_j$  at depth  $x$ . In the calculations the modified Kinchin-Pease formula was used as  $\gamma(T)$ , Lindhard's differential scattering cross-section was taken as  $d\sigma/dT$ ,  $E = 29$  eV, and  $\Phi(E_i, \theta_j, x)$  was the distribution given by Bardos [18].

The inhomogeneity of defect production is not too large throughout the specimen thickness, as we worked in the transmission mode. Apparently, the defect production cross-section varies in the range of  $0.77-1.15 \cdot 10^{-18} \text{ cm}^2$ , as shown in fig.1. The homogeneity of the ion beam distribution along the gauge length of the samples was controlled by a multifoil secondary electron-emission monitor system [15]. Owing to this device the inhomogeneity did not exceed 10%.

The mean defect production rate was  $1.2 \cdot 10^{-7} \text{ at}^{-1} \text{ s}^{-1}$  (at a beam intensity of  $1.2 \cdot 10^{11} \text{ ion cm}^{-2} \text{ s}^{-1}$ ).

After the irradiations the stress-strain curve was measured at room temperature by an Instron tensile testing machine. The strain rate was  $1.1 \cdot 10^{-4} \text{ s}^{-1}$ , corresponding to a crosshead speed of 0.1 mm/min.

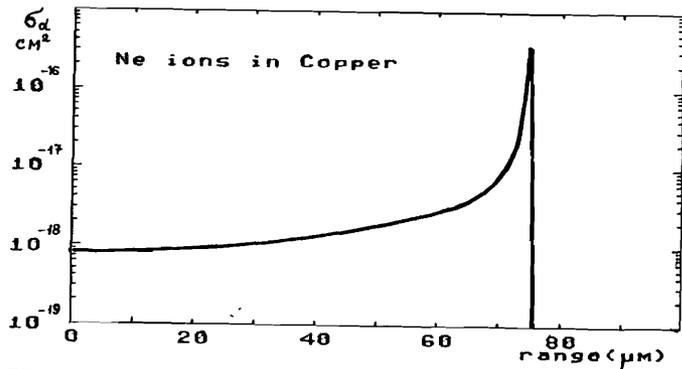


Fig.1. Frenkel-pair production cross-section vs. projectile range for 233 MeV Ne ions in copper.

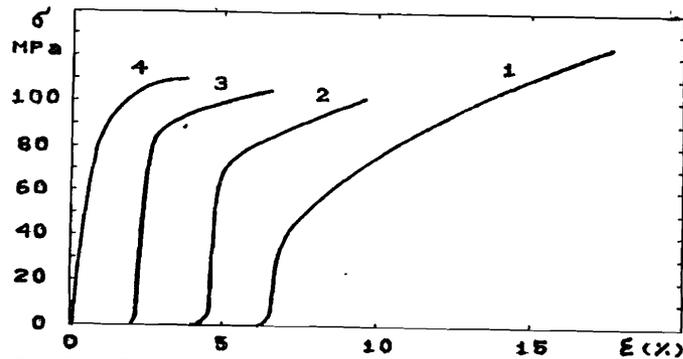


Fig.2. The stress-strain curves for copper showing their dose dependence.

1. annealed sample
2.  $\phi t = 1 \cdot 10^{15}$  ion  $\text{cm}^{-2}$ ,
3.  $\phi t = 5 \cdot 10^{15}$  ion  $\text{cm}^{-2}$ ,
4.  $\phi t = 6 \cdot 10^{15}$  ion  $\text{cm}^{-2}$ .

### 3. Results and discussion

The dose dependence of some representative stress-strain curves is shown in fig.2. to demonstrate the effect of Ne ion irradiation. It can be seen that there are (a) a large increase in yield stress, (b) a large decrease in strain to fracture, and (c) the ultimate tensile strength is less influenced by the Ne ion irradiation. As a matter of fact, these effects resemble the results for nickel irradiated by energetic heavy ions of Ne [16], and the results for neutron irradiated copper [10,20].

#### 3.1. Change in yield stress

The yield stress, according to the  $\epsilon = 0.002$  plastic deformation, was determined from the stress-strain curves. Changes in yield stress  $\Delta\sigma$  versus ion irradiation fluence ( $\phi t$ ) are shown in fig.3. At some fluences several repeated measurements have been performed to demonstrate the uncertainty in data, which does not exceed  $\pm 3$  MPa according to fig.3.

The starting value of the yield stress, measured on the unirradiated specimens, was  $\sigma_0 = 35.5$  MPa.

It can be seen in fig.3 that  $\Delta\sigma$  rises monotonically saturating at the fluence  $\phi t \sim 3 \cdot 10^{15}$  ion  $\text{cm}^{-2}$ .

The yield stress increase is presumably caused by irradiation defect clustering. These clusters prevent the dislocation propagation according to the relation [22]

$$\Delta\sigma = \alpha \mu b \sqrt{cd}, \quad (2)$$

where  $\alpha$  is a constant,  $\mu$  is the shear modulus,  $b$  is the Burgers vector, and  $c$  is the cluster density with the mean diameter  $d$ .

Inasmuch as the yield stress obviously shows a saturation character, it is worth trying to apply a simple saturation model. This model is based upon the assumption that no new cluster is formed in the vicinity of the already existing one within the volume  $v$ . According to the model, the cluster concentration rate is given by

$$dc/d(\phi t) = \sigma_c(1 - cv), \quad (3)$$

where  $c$  is the cluster concentration,  $\sigma_c$  is the cluster production cross section, and  $v$  is the effective interaction volume between the defect clusters. The solution of Eq.(3) gives the dose dependence of the cluster concentration:

$$c = 1/v(1 - e^{-\sigma_c v \phi t}). \quad (4)$$

The dose dependence of the yield stress is determined from Eq.(2) incorporating Eq.(4):

$$\Delta\sigma = A(1 - e^{-B\phi t})^{1/2}, \quad (5)$$

where  $A$  and  $B$  are related to physical parameters as follows:

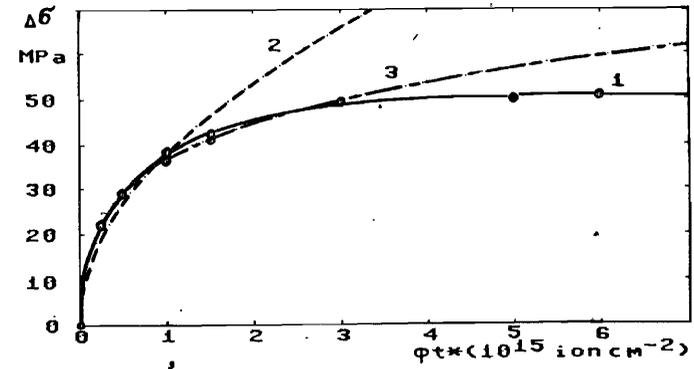


Fig.3. The yield stress vs. Ne ion irradiation fluence for Cu (●). Curves 1, 2, and 3, correspond to relations (5), (9) and (10), respectively.

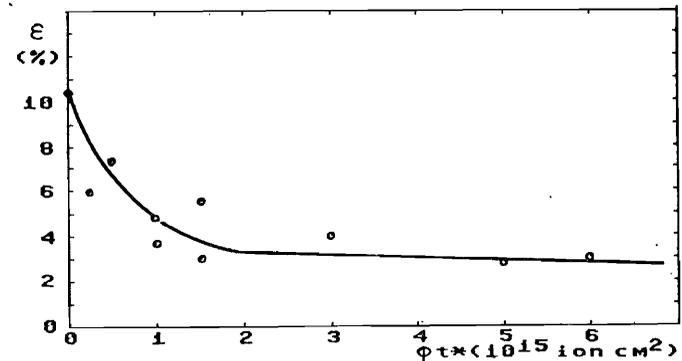


Fig.4. The deformation to fracture as a function of the Ne ion fluence.

$$A = \mu b d^{1/2} / v^{1/2}, \quad (6)$$

$$B = \sigma_c v. \quad (7)$$

Curve 1. in fig.3 demonstrates relation (5), fitted to the experimental data. There is a fairly good agreement between the data and the mathematical curve, including the saturation too. The parameters A and B, given by the least-squares method are as follows:

$$A = 51 \text{ MPa}$$

$$B = 0.8 \cdot 10^{-45} \text{ cm}^2.$$

In order to estimate the physical reality of such a model in our case, the cluster production cross-section without their interaction can be expressed in terms of microscopical parameters as follows:

$$\sigma_c = \sigma_d \xi \zeta \cdot 1/n. \quad (8)$$

The parameters incorporated in relation (8) are as follows:  $\sigma_d$  is the Frenkel-pair production cross-section,  $\zeta$  is the atomic density of copper,  $\xi$  is the defect-production efficiency, that is the ratio of defects avoiding the subthreshold annihilation to the number which theoretically is calculated according to the modified Kinchin-Pease expression [13], and  $n$  is the mean number of defects in a cluster at a low dose. To obtain an estimate of the

interaction volume  $v$ , the following actual values of the parameters were taken:  $\sigma_d = 10^{-18} \text{ cm}^2$ ,  $\zeta = 8.5 \cdot 10^{22} \text{ cm}^{-3}$ ,  $\xi = 0.5$  [13],  $n = 10$ . The evaluation leads to a prediction of  $v = 9.4 \cdot 10^{-20} \text{ cm}^3$ . Assuming the spherical form of this volume we obtain its radius to be  $r = 3 \text{ nm}$ .

It cannot be expected from such a simple model to describe the fine details of the complex interaction and annihilation processes in the high-energy (5-10 keV) displacement cascades. Nevertheless it is hoped that the main features are reflected. According to this model, the saturation is explained by the decreasing cluster concentration rate with increasing fluence, which is caused by the interaction between the already existing clusters and the newly produced cascades. The above measurements do not give information about the concrete mechanism of this interaction. However, it may be not too far from reality if it is assumed that the focussed interstitial propagation and the residual agitation of the lattice by the "thermal spike" may play an important role.

As a consequence of the interaction, no new cluster is formed, if a new cascade arises in the vicinity of an already existing cluster, within the interaction volume; however it can lead to changes in the dimensions of the already existing cluster depending on the type of the interacting clusters. In extreme cases the old cluster can disappear at all. There is experimental evidence for such annihilation of clusters [19]. In this sense the given interaction volume ( $9.4 \cdot 10^{-20} \text{ cm}^3$ ) is physically acceptable, but it is clear that it is an effective volume resulting from more concurrent processes.

It is important to note that the model presented here implies that the mean diameter of the clusters is the function of fluence, that is  $d=d(\Phi t)$ . However it can be realised, that  $d(\Phi t)$  is a slowly altering function, and it is so for  $d^{1/2}$  to a greater extent. Consequently the exponential term dominates in the yield stress changes, and it gives the saturation character.

Some comments must be made at this point.

a/ The majority of the earlier measurements for neutron irradiated copper gave another dose dependence of the yield stress  $\Delta\sigma$ . In order to compare with our data, two of them are given in fig.3. Curve 2 is plotted according to the relation

$$\Delta\sigma = e(\Phi t)^{1/2}, \quad (9)$$

as proposed by Seeger [21]. It can be seen in the figure, that whereas the first points are lying on the curve, it rises much more rapidly than the experimental data.

The Curve 3 shown in the same figure follows from the relation

$$\Delta\sigma = f(\Phi t - g)^{1/2}, \quad (10)$$

proposed by Zinkle and Kulcinski [5]. A small incubation dose is supposed by us in formula (10), for the sake of a better fit. It is seen that a rather good agreement can be obtained except for the saturation region, starting at about  $3 \cdot 10^{15}$  ion  $\text{cm}^{-2}$ . The parameters  $e, f$  and  $g$  in expressions (9) and (10) are constant.

b/ Makin and Minter [20] have used a model similar to the one presented above, explaining their measurements on neutron irradiated polycrystalline copper and nickel. Their interpretation however differs from ours.

c/ The problem of incubation fluence was examined in a direct way too. The yield stress of an unirradiated specimen was measured by deforming it only slightly over the yield point, and then releasing it. In this condition the specimen was irradiated by a fluence of up to  $0.05 \cdot 10^{15}$  ion  $\text{cm}^{-2}$ , which is less than the incubation fluence predicted by relation (10). The jump in stress observed at the subsequent deformation indicates the absence of incubation. It is worth mentioning that relation (5) does not imply an incubation dose.

d/ The role of the already existing dislocation lines is not considered at all, although they can influence first of all the formation of the interstitial clusters.

### 3.2. Other parameters of the stress-strain curve

Fig.4. demonstrates the change of strain to fracture vs. ion irradiation fluence. It can be seen that there is a rapid decrease in data starting from  $\epsilon=0.1$  down to  $\epsilon=0.03$ , which is similar to the trend observed in the case of Ne ion irradiated nickel [16].

The ultimate tensile stress has another character and strongly differs from the behavior of that of nickel [16]. At the outset of the curve it falls, then passes through a minimum and then slowly rises again (fig.5.).

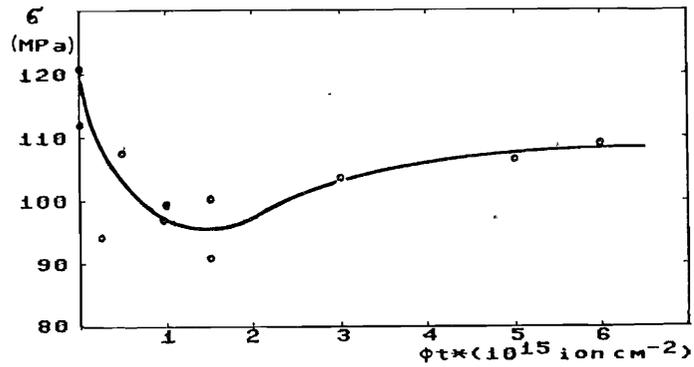


Fig.5. The ultimate tensile stress as a function of the Ne ion fluence.

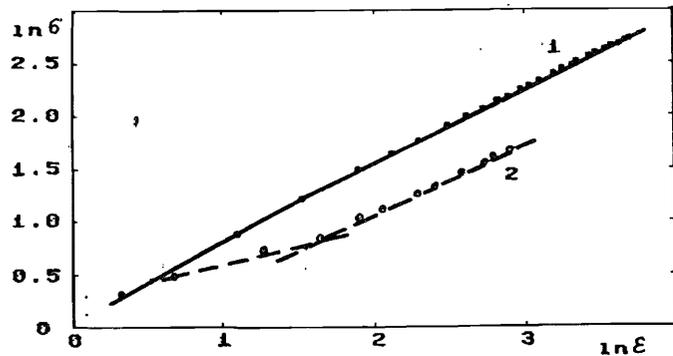


Fig.6. The  $\ln \epsilon - \ln \sigma$  plot for an unirradiated (1) and irradiated (2) copper sample.

The functional form of the stress-strain curves over the yield point was examined too. As it is usually suggested in the literature,

$$\sigma = K \epsilon^m, \quad (11)$$

where  $\sigma$  and  $\epsilon$  are the plastic parts of the stress and strain, respectively,  $K$  is a constant, and  $m$  is the work hardening coefficient. In order to determine the coefficient  $m$ , the  $\ln \epsilon - \ln \sigma$  plots were made, as shown in Fig.6. It consists of straight lines which confirm the validity of relationship (11). In the case of an unirradiated sample there is a single straight line, while for the irradiated specimens a breakpoint is observed, that is two different values of the work hardening coefficient  $m_1$  and  $m_2$  belong to one fluence. As shown in fig.7,  $m_1$  and  $m_2$  behave differently vs. fluence. At low fluences  $m_1$  rapidly falls, then passes through a minimum at about  $1.5 \cdot 10^{15} \text{ ion cm}^{-2}$ , and rises to the starting value. This trend resembles that of ultimate tensile stress. At the same time, at low doses  $m_2$  seems to remain unaffected and then it starts decreasing at a fluence of about  $1.5 \cdot 10^{15} \text{ ion cm}^{-2}$ .

The results obtained obviously demonstrate that there are two well separated parts of the deformation of the irradiated specimen, that is the mechanism of deformation changes at a certain point of elongation. The first of them may be associated with the propagation of the Luders band. This assumption is confirmed by the observation that the stress-strain curves have a jerky character immediately above

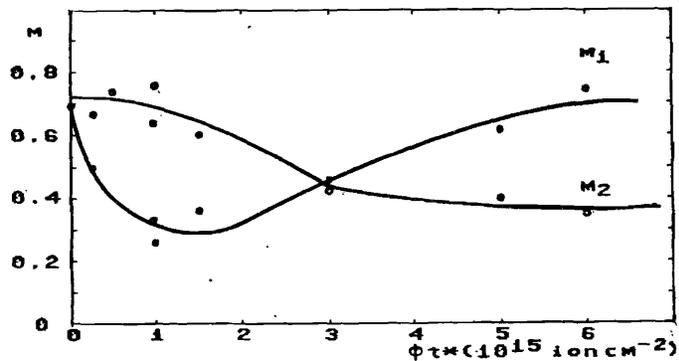


Fig.7. The dose dependence of the work hardening coefficients  $m_1$  and  $m_2$ .

the yield point, which is likely to be the consequence of inhomogeneous deformation.

At low fluences the coefficient  $m_1$  remains almost unchanged. Similar observation has been reported by Higgy [10] for pure copper single crystals after a high neutron exposure, and by Makin and Minter for neutron irradiated polycrystalline copper [20]. As far as the ultimate tensile stress, the deformation to fracture and the work hardening coefficient are concerned, the uncertainties in their measurement are usually greater than those of the yield point, and their features are not easy to interpret without additional measurements.

#### 4. Acknowledgements

The authors wish to express their gratitude to Academician G.N. Flerov and Prof. Yu.Ts. Oganessian for their permanent interest, as well as to G. Bardos for the defect production cross section calculations. We are indebted to the personnel of the cyclotron U-400 for their careful work.

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Влияние облучения ионами неона  
с энергией 233 МэВ на механические свойства меди

Исследовалось радиационное упрочнение меди, облученной ионами неона с энергией 233 МэВ. Показано, что имеет место возрастание предела текучести. Дозовая зависимость имеет вид монотонно возрастающей кривой с насыщением при дозе  $3 \cdot 10^{15}$  ион/см<sup>2</sup>. Результаты объясняются на основе модели, в которой предполагается взаимодействие образующих кластеров.

Работа выполнена в Лаборатории ядерных реакций ОИЯИ.

Препринт Объединенного института ядерных исследований. Дубна 1987

Havancsak K. et al.

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233 MeV Ne Ion Irradiation Effects on the  
Mechanical Properties of Copper

The dose dependence of the deformation behaviour of copper irradiated by 233 MeV Ne ions was investigated. The experiments were carried out on an external beam of the U-400 cyclotron. The yield stress rises monotonically from  $3 \cdot 10^{14}$  ion cm<sup>-2</sup> dose, saturating at  $\phi t \sim 3 \cdot 10^{15}$  ion cm<sup>-2</sup>. This saturation behaviour is described by a simple saturation model, which is based upon the assumption that the cluster concentration rate decreases with increasing fluence. It is assumed that this is caused by the interaction between the already existing clusters and the newly produced cascades. The changes of other parameters of the stress-strain curve are given too.

The investigation has been performed at the Laboratory of Nuclear Reactions, JINR.

Preprint of the Joint Institute for Nuclear Research. Dubna 1987