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DEFECTS AND TRANSMUTATIONS IN REACTOR-IRRADIATED COPPER**R. L. Chaplin****CLEMSON UNIVERSITY, CLEMSON, SOUTH CAROLINA****and****R. R. Coltman, Jr.**

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DEFECTS AND TRANSMUTATIONS IN REACTOR-IRRADIATED COPPER*

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ABSTRACT

From measurements made at 4.2 K, the residual resistivity increases produced by reactor irradiation near room temperature were studied in pure Cu up to fluences of $4.2 \times 10^{23} \text{ n/m}^2 > 1 \text{ MeV}$ and $12 \times 10^{23} \text{ thermal n/m}^2$ by methods which resolved the effects due to transmutations and to defects. While the increase in resistivity due to transmutations is linear, that due to defects falls off rapidly with increasing fluence. The defect damage rates at low doses depend upon initial sample conditions that are related to impurities, but they become equal above $\sim 6 \times 10^{21} \text{ n/m}^2$ ($E > 1 \text{ MeV}$). Computations of defect concentrations using these resistivity data and earlier x-ray results [1], which measure dislocation loops, can be brought into reasonable agreement if the specific resistivity attributed to a single defect is decreased when that defect becomes part of a dislocation loop. In addition to the fundamental interest of these results, they also suggest that during the life of certain water-cooled Cu magnets for fusion reactors the resistivity increases due to defects will be small compared to those produced by transmutations.

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DEFECTS AND TRANSMUTATIONS IN REACTOR-IRRADIATED COPPER

R. L. Chaplin and R. R. Coltman, Jr.

1. INTRODUCTION

Studies of the increase in electrical resistivity of Cu produced by irradiation near room temperature are of interest for both the fundamental understanding of defect behavior in metals and the near-future practical needs of fusion reactor technology. This report relates to both areas by giving information derived from measurements of changes in the residual resistivity of Cu made after each of many successive reactor irradiations.

Extensive studies of damage production in Cu have already been made near 4 K and throughout the stage-II temperature range, using many types of irradiation. In contrast, few experiments have dealt with reactor irradiation near room temperature. Such studies require samples to be cooled to liquid-He temperature to make accurate residual-resistivity measurements, and the presence of radioactivity necessitates either special handling techniques or long waiting periods for radioactive decay as done by Blewitt et al. [2]. In the present work, samples were transferred by remote handling techniques into a shielded liquid-helium cryostat a short time after a reactor irradiation near room temperature. This procedure enabled changes to be followed on individual samples throughout the course of the irradiation study with an accuracy much greater than that of earlier studies [3,4], which measured resistivity changes during reactor irradiation at ambient temperature.

X-ray studies of damage produced in copper in the same irradiation facility as that used in this work [1, 5] show the presence of both interstitial and vacancy dislocation loops. That analysis suggests that both

types of defects are mobile at room temperature. Our results can be correlated with the x-ray data above a fluence of $1 \times 10^{22} \text{ n/m}^2$ ($E > 1 \text{ MeV}$). Below this fluence, the results indicate the presence of defect traps, which are active at room temperature in copper and influence the retention of defects.

2. EXPERIMENT

Two types of wire specimens (25 mm long x 0.25 mm diameter) were prepared in pairs in accordance with the annealing treatments listed in Table I. Data presented in the table show that the smallest residual resistivity, ρ_0 , was observed for sample pair #1, and this result is attributed to the internal oxidation and precipitation of impurities by the oxidizing anneal. The high-vacuum anneal given to pair #2 is considered a reducing treatment.

A cylindrical sample holder was fabricated from white lavite, and one of each of the two types of samples was mounted in the upper and lower sections of the lavite. The lower half of the holder had inner and outer concentric cylinders of cadmium foil which shielded the two lower samples from thermal neutrons without interfering with the fast-neutron flux. Four wires were soldered to each sample for making standard potentiometric measurements at times when samples were cooled to 4.2 K in the liquid-helium cryostat. The stability and reproducibility of the equipment gave a sensitivity of approximately $3 \times 10^{-14} \Omega \text{ m}$ for electrical-resistivity determinations.

The irradiation facility situated in the pool-type Bulk Shielding Reactor (BSR) at Oak Ridge National Laboratory provides at nominal full power (2 MW) a thermal-neutron flux, ϕ_t , of $13 \times 10^{16} \text{ n/m}^2 \text{ sec}$ and a

fast-neutron flux, ϕ_f , of 4.4×10^{16} n/m² sec. ($E > 1$ MeV) [6]. Although the BSR pool water is maintained at a temperature of 34°C, nuclear heating accounts for temperatures of the samples which approach 60°C during an irradiation. Samples were subjected to many successive neutron irradiations ranging from 2 hours to 3 weeks. The thermal- and fast-neutron fluence increments, Φ_t and Φ_f , for each irradiation were found from the aforementioned flux values and the integrated reactor power. Generally after an irradiation, the radioactive sample assembly was allowed to decay for a minimum of 12 hours to eliminate short-lived activity before samples were transferred by remote handling to the shielded cryostat.

3. RESULTS AND DISCUSSION

3.1 The Separation of Defect and Transmutation Effects

Virtually all of the increase in residual resistivity produced in copper and other metals by reactor irradiation arises from the action of thermal and fast neutrons. Fast neutrons produce cascades of lattice defects as a result of direct elastic collisions with host atoms. Thermal neutrons can also produce defects by their capture and the resulting (n, γ) reaction [7], but the effect of these low-energy events was not detected in this experiment. In addition, thermal neutrons also produce Zn and Ni transmutations in Cu.

Previous measurements of the spatial dependence of the fast-neutron flux in the irradiation facility [6] show, that for the relatively small separation between the Cd-shielded and unshielded specimen partners, both units receive virtually the same fast-neutron flux. In contrast, the thermal-neutron flux at the Cd-shielded specimens is much less than that

at their unshielded partners. For this reason, the same external thermal-neutron flux produced resistivity increases due to transmutations, ρ_T , which were greater for the unshielded samples.

The measured resistivity increase for a particular combined fast- and thermal-neutron fluence can be written as

$$\rho_{TOT} = \rho_D + \rho_T, \quad (1)$$

where the subscripts D and T refer to defects and transmutations, respectively. The above equation is valid for both shielded and unshielded specimens, so henceforth, superscripts S and U will denote quantities relating to shielded and unshielded samples.

As stated above, experimental conditions insure that $\rho_D^U \equiv \rho_D^S$, so when this equality is combined with equation (1), it follows that

$$\rho_{TOT}^U - \rho_{TOT}^S = \rho_T^U - \rho_T^S. \quad (2)$$

Since in Eq. (2) both ρ_T^U and ρ_T^S are unknown (ρ_{TOT}^U and ρ_{TOT}^S are measured quantities), additional information is needed to solve the equation. This was supplied by the results of a corollary experiment which measured the quantity

$$\frac{\rho_T^U}{\rho_T^S} \equiv r \quad (3)$$

where r is a constant. The value of r was determined for each sample pair early in the irradiation history of the experiment. This was accomplished by measuring immediately after a 12-hour irradiation the residual-resistivity increases which occurred as a result of radioactive decay. This type of resistivity increase, which has been studied previously in detail for several elements [8], arises from the transmutation and damage

effects which occur during decay of a radioisotope. In the present case ^{64}Cu is produced during the irradiation by neutron capture in ^{63}Cu (69% abundance). The ^{64}Cu decays with a 12.8-hour half-life by both β^+ and β^- emission to produce ^{64}Zn and ^{64}Ni , respectively. The resistivity increase in each sample is then directly proportional to only the thermal-neutron fluence it received, and, therefore, the ratio of the resistivity increases in the partners of a pair gives the value of r .

By combining Eqs. (1), (2), and (3) it is then possible to obtain the desired quantities in terms of measured quantities:

$$\rho_T^U = \frac{r(\rho_{TOT}^U - \rho_{TOT}^S)}{r - 1}$$

$$\rho_D^U = \rho_D^S = \rho_{TOT}^U - \rho_T^U = \rho_{TOT}^S - \rho_T^S$$

To complete the analysis, values of each of the desired quantities can be plotted against values of the appropriate neutron fluence which corresponded to the ρ_{TOT} values. The fluence dependence of ρ_{TOT}^U , ρ_T^U , and ρ_D^U are given in Figure 1 for sample pair Number 1. Differences in the response of ρ_D to Φ_f between the two sample pairs are discussed below. It is interesting to note that the responses of ρ_T to Φ_t between the two sample pairs agreed to better than 1%. Since the initial resistivities of the sample pairs differed by nearly a factor of 4, this result suggests close adherence to Matthiessen's rule.

Using handbook values for the nuclear properties of Cu, we calculate the transmutation concentrations in the unshielded specimens to be 240 ppm each of Zn and Ni for the highest fluence reached. It has long been known from low-temperature-damage-recovery studies of Cu doped with much higher concentrations of these elements that these solutes have virtually no

interaction with irradiation defects [9]. For this reason, we believe that the transmutations are not active defect traps, and they have no significant influence on the damage-retention behavior in this experiment.

3.2 The Retention of Defects

By means of x-ray diffuse scattering, Larson and Young have studied the effect of reactor irradiation on the growth of dislocation loops in copper [1] irradiated in the same facility as that used in this experiment. At the lowest fluence studied in the x-ray work [1×10^{22} n/m² ($E > 1$ MeV)] they found that loop structures had already been established. The loops, which are produced by the collapse of defect clusters, were identified as both vacancy and interstitial types having equal populations. The x-ray studies determined the number and size distribution of loops up to a fluence of $\sim 1 \times 10^{23}$ n/m² ($E > 1$ MeV). The present resistivity studies, which were carried out under virtually the same irradiation conditions, spanned over the x-ray range to both higher and lower fluence values. The presentation and discussion of the resistivity results given in this section attempt to explore any correlation with the x-ray results. In addition, the discussion is based on the commonly held view that a fast-neutron-produced displacement cascade yields small vacancy and interstitial clusters, and probably some as small dislocation loops which are stable at room temperature, along with single and possibly double defects which are mobile. While the mobile defects account for losses by recombination, they also promote cluster and loop growth by reactions with clusters and loops of like type.

In addition to their measurements of the distribution of loop sizes, Larson and Young [1] also determined total defect concentrations as a

function of fluence. This was accomplished by counting lattice sites encompassed by both vacancy and interstitial loops; i.e., retained defect concentrations were proportional to all area encompassed by loops. From their data, we have calculated the equivalent Frenkel-pair production rates (using half their concentration values) and compared them with rates calculated from the resistivity data. The latter were determined from the data in Fig. 2, which show for both sample pairs the damage rate vs damage resistivity, ρ_D , for the full range of fluence studied. Resistivity damage-rate values taken from the fluence range spanned by the x-ray data were converted to Frenkel-pair concentrations using the well-known specific Frenkel-pair resistivity value, $\rho_F = 2 \times 10^{-6} \Omega \text{ m/unit fraction}$. The x-ray and resistivity results are compared in Fig. 3, which shows two main features:

1. Throughout the fluence range studied in the x-ray experiment, the ratio of the defect-production rates (right ordinate of Fig. 3) determined from resistivity and x-ray measurements is constant within experimental accuracy. In further support of the correlation found here during defect production, Larson also found a one-to-one correlation between x-ray and resistivity measurements made during the annealing of reactor-irradiated copper [5]. These results suggest that the resistivity changes are proportional to changes in area encompassed by the dislocation loops and hence to the defect concentrations related therein. However, since the size distribution of the loops as well as the loop concentrations changed with neutron fluence, or with annealing, it is not clear whether the cores of the dislocation loops make a significant contribution to the resistivity change.

2. The comparison in Fig. 3 shows that the constant ratio of x-ray to resistivity-determined defect concentrations is 3.2. While the x-ray and resistivity measurements show a qualitative correlation during both damage production and annealing, this quantitative mismatch has been a puzzle for some time.

Transmission-electron-microscopy studies [10] of copper that was neutron irradiated under conditions similar to these experiments have shown that both vacancy and interstitial loops are formed by the irradiation, and that the great majority of these loops have Burgers vectors $a/3 \langle 111 \rangle$; i.e., they are faulted. There is little evidence of stacking-fault tetrahedra. If it is assumed that all loops are faulted and that the total stacking-fault area is responsible for the measured increase in resistivity (we assume that the concentration of point defects is negligible), then the expected resistivity change can be calculated.

The change in resistivity caused by stacking faults is defined as $\Delta\rho_s \equiv \rho_s \beta$, where ρ_s is the resistivity introduced per unit area of stacking fault and β is the area of stacking fault per unit volume. Howie [11] has calculated the value $\rho_s = 1 \times 10^{-16} \Omega \text{ m}^2$ for copper. We will assume that this value is correct for both intrinsic (vacancy loops) and extrinsic (interstitial loops) stacking faults. The numerical values for β can be computed directly from the point-defect concentrations reported by Larson and Young [1]. The area per atom in copper $\{111\}$ planes is $\sim 5.4 \times 10^{-20} \text{ m}^2$. For the neutron fluence of 1×10^{22} and $1.1 \times 10^{23} \text{ n/m}^2$ Larson and Young found point-defect concentrations 1.25 and $7 \times 10^{25}/\text{m}^3$, respectively. The computed $\Delta\rho$'s are 0.067 and $0.38 \text{ n}\Omega\text{m}$ for the respective fluences, to be

compared with our measured resistivity changes of 0.068 and 0.29 for identical respective fluences. This is rather good agreement.

We have also calculated the resistivity change that might be expected from the dislocation lines (cores) themselves. Basinski et al. [12] have measured the resistivity (as $\Omega \text{ m}$ per meter of dislocation line per m^3) of slip dislocations in copper; $\rho_d = 1 \times 10^{-21} \Omega \text{ m}^3$. We assume that the value of ρ_d for $\underline{b} = a/2 [110] \approx \rho_d$ for $\underline{b} = a/2 [111]$, with no other contributions to the resistivity. Then the computed $\Delta\rho_d$ values were about 0.06 and 0.3 $\text{n}\Omega\text{m}$ for the two neutron fluences cited above. For this comparison we took average loop radii to be 15 Å and 20 Å for the respective fluence values. A more precise calculation for $\Delta\rho_d$ values would require use of the exact measured size distribution, but the error introduced by using these average loop sizes is small compared to the other assumptions. Again, agreement between measurement and calculation is good.

We conclude that it is not possible with the available data to determine the source of the measured resistivity changes. Either the stacking faults or the dislocation cores can give reasonable numerical agreement. It remains unclear as to why the ratio of point defects as measured by diffuse scattering of x rays to that as measured by change in resistivity is about 3. It is possible that this "constant" is a fortuitous one, holding only for the particular range of loop concentration and size distribution existing in these experiments.

So far in this section we have discussed the retention of defects for fluences greater than $1 \times 10^{22} \text{ n/m}^2 > 1 \text{ MeV}$ at which loops were first observed by x-ray studies [1]. We now focus attention on the range of fluence below this value.

Figure 4 shows both the production (upper curves) and the incremental production rates (lower curves) of damage resistivity for relatively small fluences. It can be seen that in this range the retention of defects depends upon the preirradiation sample conditions. Initially, the higher damage rate (40% higher at the first data point) occurs for sample pair No. 2 with the higher initial resistivity. It can also be noted that with increasing fluence the dependence of damage rate upon initial sample condition is steadily reduced. At a fluence of $\Phi_f \sim 6 \times 10^{21} \text{ n/m}^2 > 1 \text{ MeV}$ the rates become equal (within experimental error). These results are consistent with the commonly held view of fast-neutron displacement cascades mentioned in the beginning of this section; at first, mobile defects are retained at active impurity traps in the samples whose concentrations are related to the initial sample resistivities. Then, the growing population of neutron-produced stable defect clusters provides a steadily strengthening competition with impurities for the mobile defects until the clusters finally dominate mobile defect reactions. Reactions between mobile defects and stable clusters can, of course, produce defect recombinations as well as add to cluster growth.

The defect behavior just described is virtually the same in principle as that which operates during electron irradiation in the Stage-II temperature range where only the interstitial is mobile. The correspondence is achieved by the fact that, although both vacancies and interstitials are mobile at room temperature, the fast neutrons produce small immobile defect clusters which play a role corresponding to the immobile vacancies in Stage II. Thus, at low fluences impurity trapping, defect recombination, and cluster growth operate during fast-neutron irradiation at room temperature.

Accordingly, one might expect the present results to show the well-known linear relationship between reciprocal damage rate and damage resistivity observed for electron irradiation in the Stage-II temperature range [13]. This possibility can be examined in Fig. 5, where the low-fluence data are plotted in this fashion.

In plots like Fig. 5, Stage-II electron-irradiation studies typically show straight lines originating at a common ordinate value and with slopes dependent upon initial impurity content. For the data in Fig. 5 it is not clear as to whether a common ordinate value exists. Studies at smaller doses than were possible here are needed to make such a judgment. Further, instead of straight lines both sample pairs show an "S"-shaped response in this plot. Another view of this behavior is seen in Fig. 4 where, initially, the decrease in the damage rate is very rapid and then quickly slows to give an elbow-like appearance to the curve. This response is consistent with the idea that in this fluence range defect clusters have begun to collapse into dislocation loops where the defect resistivity is reduced. Thus, over this fluence range the average specific resistivity of the defects is being reduced as a result of this change in configuration. The resistivity data, then, reflect a greater decrease in damage rate than is actually occurring. It is important to note in this scheme that when the addition of a new defect stimulates collapse of the cluster, not only the resistivity of the new defect but that of all members of the cluster is reduced. Overall, we do not see a resemblance between the electron and neutron cases as was expected above.

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3.3 The Impact of Irradiation Resistivity on Designs for Fusion-Reactor Magnets

Along with the large superconducting toroidal-field coils which provide plasma containment in a tokamak fusion reactor, additional coils are needed to serve other functions [14]. While the superconducting coils will be shielded to provide some protection against radiation damage, certain inner poloidal-field (PF) coils will be virtually unshielded except for the intervening first wall and blanket. It has been considered that these coils may be constructed of copper and cooled by water near room temperature to remove nuclear and eddy-current heating. The data given in this report, which were obtained under temperature conditions similar to those of a PF coil, provide some insight into the radiation-damage effects to be expected in such a coil.

Santoro et al. [15] have calculated the neutron spectra and intensities throughout a reference design experimental power reactor [14] having a neutron wall loading of 1 MW/m^2 . For our needs in this report, Santoro provided us in computer-output form certain neutron spectra and other pertinent data obtained during the course of his studies, but not reported in ref. [15]. This allowed us to compare neutron conditions at the PF coil with those in the facility used for this experiment [6].

From his results, we found that at the PF coil that 96% of the neutron population will be below 6 MeV, and that portion of the spectrum was much softer (smaller fraction of the neutrons at higher energies) than ours. The remaining 4% of the PF spectrum produced a high energy tail extending to 14 MeV which was considerably harder than ours. The damage-energy cross-section determined by Santoro using the RECOIL code [15] was $\langle \sigma T \rangle = 22 \text{ keV}$

barns. For our spectrum it is sufficient for the comparison which follows to assume a fission spectrum where $\langle \sigma T \rangle = 82 \text{ keV barns}$ [16]. If we assume for the moment that the damage efficiencies [16] of the spectra are equal, then the damage produced by unit fluence in our facility is $82/22 = 3.7$ times greater than that at the PF coil. The annual neutron fluence, Φ_A , at the PF coil is calculated to be $1.25 \times 10^{25} \text{ n/m}^2$ (average of two values for different absorber and coolant materials in the blanket). For comparison, the highest fluence reached in our experiment was $4 \times 10^{23} \text{ n/m}^2$ ($E > 1 \text{ MeV}$), or $1.2 \times 10^{24} \text{ n/m}^2$ ($E > 0.1 \text{ MeV}$) when all neutrons are counted which produce significant damage. If our fluence value is adjusted upward by the ratio of damage-energy cross-section, it becomes $4.4 \times 10^{24} \text{ n/m}^2$ or equivalent to about 1/3 year of coil operation. If the damage efficiency of the PF coil spectrum is greater than that for our spectrum, as we suspect, then the equivalent time could be even less than 1/3 year.

For our maximum dose we found a resistivity increase at 4.2 K of $0.48 \text{ n}\Omega\text{m}$ for all samples. This increase was also measured at room temperature and found to exceed the low temperature value by a factor of 1.5, indicating a deviation from Mattheissen's rule (DMR) when phonon resistivity was added to defect resistivity. In terms applicable for magnet design we observed a resistivity increase at room temperature of $0.72 \text{ n}\Omega\text{m}$ or close to 4% for our maximum dose. At this dose, however, the resistivity-damage rate was reduced to only about 2% of initial values. If we assume the unlikely possibility of no further decreases in the rate and apply our lowest measured rate for a possible lifetime fluence equivalent to 50 times our maximum dose, then we calculate a further increase of only $0.08 \text{ n}\Omega\text{m}$, which when added to our highest observed resistivity

gives a total of 0.80 n Ω m. Thus, from our results we estimate a lifetime resistivity increase for copper in the poloidal field coil of no more than 5% at room temperature due to retained irradiation effects.

In addition to the experimentally determined estimate of defect resistivity, that due to transmutations was also calculated. First, activation cross-sections, σ_a , of Cu for the nuclear reactions listed in Table II were determined using Santoro's PF coil spectrum. Then resistivity values were found by $\rho_T = \sigma_a \Phi_A \Omega_T$ where Ω_T is the specific resistivity of the transmuted species in Cu.

By comparing the resistivity increases due to defects and transmutations it is noted that the defect resistivity is higher during the first year. After 20 years of operation, however, the transmutation resistivity, which increases linearly with fluence, reaches 6.6 times the defect resistivity, and the total becomes 35% of the initial room-temperature value (the same DMK value found for defects was applied to transmutations).

4. CONCLUSIONS

1. The determination of defect concentrations in neutron-irradiated copper by x-ray and resistivity methods can be brought into reasonable agreement if the specific resistivity of the dislocation loops and/or stacking faults are considered; the resistivity change attributable to a single defect is decreased when the defect becomes a part of a dislocation loop.

2. To analyze defect retention behavior at low neutron fluences using resistivity data, consideration must be given to the fact that the average specific resistivity of defects decreases as a result of the collapse of defect clusters into dislocation loops.

3. Over the lifetime of poloidal field coils used in large tokamak fusion reactors, it is expected that the increase in electrical resistivity due to transmutations will be several times greater than that due to retained defects.

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Table I. Preirradiation Specifications

Sample Pair No.	Annealing Treatment	Resistivity (n Ω m)		Approximate Resistivity Ratio $\rho_{300}/\rho_{4.2}$
		ρ_o^s (Shielded)	ρ_o^u (Unshielded)	
1	ASR; 5 - 9's Cu; 2 hr.; 1000°C; 10 ⁻⁴ Torr air	0.008,34	0.008,85	2000
2	ASR; 5 - 9's Cu; 2 hr.; 1000°C; 3 x 10 ⁻⁶ Torr	0.050,4	0.059,3	500

Table II

Resistivity Increase at the PF Coil for Transmutations Produced by an Annual Fluence, $\Phi_A = 1.25 \times 10^{25} \text{ n/m}^2$

Nuclear Reaction	Transmuted Species	Effective* Activation Cross-Section, σ_a (mb)	Specific Resistivity Ω_T ($10^{-6} \Omega\text{m/unit frac.}$)	Transmutation Resistivity, ρ_T (n Ω m)
(n, γ)	^{64}Zn , ^{64}Ni , ^{66}Zn	169	0.77 [7]	0.163
(n,2n)	^{62}Ni , ^{64}Ni , $^{64}\text{Zn}^\dagger$	6	1.25	0.009
(n,p)	^{63}Ni	1.3	1.25	0.002
(n, α)	^{60}Co , $^{62}\text{Ni}^\dagger$	0.3	6.3	0.002
TOTAL				0.176

*Account was taken of isotopic abundance and certain branching ratios to produce effective σ_a values for natural Cu.

† Negligible resistivity contribution.

FIGURE CAPTIONS

- Fig. 1. The total residual-resistivity increase versus thermal- and fast-neutron fluence for sample pair No. 1. The resolved contributions due to damage and transmutation resistivity are shown for comparison.
- Fig. 2. The dependence of damage rate upon damage resistivity for two differently prepared copper samples.
- Fig. 3. A comparison of X-ray- and resistivity-determined defect production rates in reactor-irradiated copper.
- Fig. 4. Damage resistivity (upper) and damage rate (lower) versus fast-neutron fluence for two differently prepared copper samples.
- Fig. 5. Reciprocal damage rate versus damage resistivity. The dotted lines are linear extrapolations to the ordinate. It is recognized that other response to a common ordinate value might be possible.

FIG. 1

FIG. 2

FIG. 3

FIG. 4

FIG. 5