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INFLUENCE OF TRANSMUTATION AND HIGH NEUTRON EXPOSURE ON MATERIALS USED IN FISSION-FUSION CORRELATION EXPERIMENTS

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INFLUENCE OF TRANSMUTATION AND HIGH NEUTRON FLUENCE ON MATERIALS USED IN FISSION-FUSION CORRELATION EXPERIMENTS

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ABSTRACT

This paper explores the response of three different materials to high fluence irradiation as observed in recent fusion-related experiments. While helium at fusion-relevant levels influences the details of the microstructure of Fe-Cr-Ni alloys somewhat, the resultant changes in swelling and tensile behavior are relatively small. Under conditions where substantially greater-than-fusion levels of helium are generated, however, an extensive refinement of microstructure can occur, leading to depression of swelling at lower temperatures and increased strengthening at all temperatures studied. The behavior of these alloys is dominated by their tendency to converge to saturation microstructures which encourage swelling.

Irradiations of nickel are dominated by its tendency to develop a different type of saturation microstructure that discourages further void growth. Swelling approaches saturation levels that are remarkably insensitive to starting microstructure and irradiation temperature. The rate of approach to saturation is very sensitive to variables such as helium, impurities, dislocation density and displacement rate, however. Copper exhibits a rather divergent response depending on the property measured. Transmutation of copper to nickel and zinc plays a large role in determining electrical conductivity but almost no role in void swelling. Each of these three materials offers different challenges in the interpretation of fission-fusion correlation experiments.

INTRODUCTION

In both the current and previous workshops in the series,[1,2] many of the papers discussed experiments that involved low dose levels achieved at low

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displacement rates. Such studies are usually directed toward the influence of PKA recoil spectra in defining the neutron spectral dependence of various damage processes. At these damage levels transmutations are of no importance. Studies involving transmutation, either gaseous or solid, usually proceed at higher displacement rates to higher dpa levels.

There is an implicit expectation in low exposure studies that the measured defect survivabilities and damage efficiencies will be applicable to more complicated damage mechanisms occurring at the higher damage rates relevant to fission and fusion reactor conditions. It is becoming apparent, however, that damage efficiencies are not fixed but evolve, first with increasing displacement rate and then in response to the consequences of earlier damage and transmutations. In effect, the microstructure begins to take charge and it eventually responds to additional considerations that have nothing to do with PKA recoil spectra. One consequence of this situation is that irradiated metals tend to forget their starting state and develop equilibrium microstructures dictated primarily by alloy composition, irradiation temperature, displacement rate and possibly the accumulation of transmutation products. Recoil spectrum considerations become secondary in such a situation.

This leads to a curious paradox where some low-dose neutron and charged particle studies reached the conclusion that defect survivabilities are on the order of 1% or less. It has been shown, however, that the intrinsic steady-state swelling rate of austenitic stainless steels is ~1%/dpa over a wide range of composition, temperature and displacement rate.[3] This represents a 1% rate of accumulation of vacancies in voids alone, and it is generally accepted that many more vacancies are accepted at dislocations. Unless the bias of dislocations toward accepting interstitials is infinite, the defect survivabilities at high fluence must be much larger than 1%. Obviously, the evolving microstructure plays a strong role in determining the fraction of defects that survive to create a given property change.

It has also been shown that fission-fusion correlation efforts directed toward the influence of either PKA recoil spectra or helium/dpa ratio are often overwhelmed by the unavoidable introduction of third variables.[4] Displacement rate differences in particular were shown to often dominate fission-fusion correlation experiments and to distort the operation of mechanisms associated with helium effects.

To demonstrate the complexity involved in correlation efforts at high dpa levels, the status will be reviewed of three high fluence studies, each on a different material and each directed toward a different spectral-related consideration. Each demonstrates that irradiated materials tend to converge to microstructural states that may not be as responsive to PKA spectral and helium/dpa considerations as might have been originally anticipated. In the following sections we will examine the response of Fe-Cr-Ni alloys, pure nickel and copper-base alloys.

He/dpa EFFECTS IN Fe-Cr-Ni ALLOYS

In a comprehensive effort to study the separate and synergistic effects of helium and other important variables involved in neutron irradiations of austenitic alloys, a series of closely related experiments on simple Fe-Cr-Ni alloys are in progress. The approach of these studies is to first determine in detail the major compositional and environmental sensitivities of microstructural development, as well as the associated changes in mechanical properties, of Fe-Cr-Ni model alloys irradiated in the EBR-II (Experimental Breeder Reactor-II) and Fast Flux Test Facility (FFTF) fast reactors. The roles of phosphorus, silicon and titanium are also included in these studies since precipitates formed by these elements are often invoked to play a role in the distribution and action of helium.[5] Some early results of these continuing studies are documented in other papers.[6-11] The fast reactor results are then being compared with the behavior of these alloys after irradiation in a spectrally tailored experiment in the Oak Ridge Research Reactor (ORR),[11-13] a companion nontailored experiment in EBR-II[3,7] and in an ^{59}Ni isotopic-doping experiment in FFTF.[13-17]

EBR-II and ORR Comparison

The AD-1 experiment was conducted on a series of Fe-15Cr-XNi (X = 25, 35, 45) and Fe-YCr-35Ni (Y = 7.5, 20) alloys to doses of 9.5 to 11.3 dpa in EBR-II at the relatively low helium/dpa ratios typical of fast reactors. These ratios are dependent on nickel and range from 0.66 to 1.2 appm/dpa for nickel levels of 25 to 45%. The MFE-4 experiment was conducted in ORR to doses of 12.2 to 14.3 dpa at helium/dpa ratios ranging from 27 to 58 appm/dpa. This experiment contained two additional nickel levels (X = 20, 30). For a given irradiation temperature the displacement level was independent of nickel content in EBR-II, but this was not the case in ORR, where the two step $^{58}Ni(n,\gamma)^{59}Ni(n,\alpha)^{56}Fe$ reaction that produces the large helium levels in this reactor also makes a measurable contribution to the displacement rate (see Table 1).

Table 1

Displacement and Helium Levels[(a)] in the MFE-4 Experiment in ORR

	330 and 400°C		500 and 600°C	
Composition, wt%	dpa	He, appm	dpa	He, appm
Fe-19.7Ni-14.7Cr	13.4	371	12.2	332
Fe-24.4Ni-14.9Cr	13.6	463	12.4	414
Fe-30.1Ni-15.1Cr	13.8	555	12.6	495
Fe-34.5Ni-15.1Cr	14.0	647	12.7	573
Fe-45.3Ni-15.0Cr	14.3	832	13.1	740

(a) These values were calculated using dosimetry calculations and measurements provided in Reference 25 for individual elements. Note that the dpa levels increase with nickel content, reflecting the contribution of the ^{56}Fe recoil atom during helium production.

Another significant difference between the two experiments arises from the order of magnitude difference in displacement rate, with the MFE/ORR experiment proceeding at a lesser rate than AD-1/EBR-II. Microstructural evolution is known to be sensitive to displacement rate, a consideration which complicates the comparison of the results of these two experiments.

Figure 1 compares the swelling behavior vs. nickel of these two experiments, measured using immersion density. Note that while there is no significant amount of swelling for any nickel level at 400°C in ORR at 13.4-14.3 dpa, measurable amounts of swelling were found at only 9.5 dpa in EBR-II at 395°C. Swelling in EBR-II exhibited the decreasing dependence on nickel content commonly observed in many other experiments. Figure 2 shows another feature of the EBR-II/ORR comparison. Throughout the temperature range studied the radiation-induced changes in yield strength are larger in ORR than in EBR-II, even though the swelling was in general lower. The microstructural origins of the behavior shown in Figures 1 and 2 are being sought using transmission electron microscopy.

The analysis effort has concentrated first on Fe-15Cr-XNi irradiations conducted at 400 and 500°C in ORR and at 395 and 450°C in EBR-II.[18] The EBR-II data at 450°C were reported in detail in an earlier paper.[7] The swelling in ORR and EBR-II as determined by both immersion density and microscopy showed relatively good agreement. No precipitates were observed in any alloy. As can be seen in Figure 3 the major difference between the two sets of irradiations is reflected in the cavity density. Whereas the EBR-II cavity densities exhibit the usual trends, decreasing both with irradiation temperature and nickel content, the densities reached in ORR not only increase with nickel content but reach levels that are two to three orders of magnitude larger than in EBR-II. At 400°C cavity densities in excess of 10^{17} cm^{-3} are some of the highest ever observed in reactor irradiation studies at this temperature. The cavity densities at 400°C are clearly saturating at levels that increase with nickel, but they are not as sensitive to nickel content as was observed at 500°C. As shown in Figure 4, the cavity sizes at 400°C increase with nickel content as do those at 330°C.

The width of the size distribution of the cavities in the ORR experiment at 500°C was observed to become progressively smaller as the nickel content increased, as shown in Figure 5. This contrasts with the behavior observed in EBR-II, where voids are in general larger but whose sizes are relatively independent of nickel content at a given irradiation temperature. Although the data are still being analyzed, it appears that there is some refinement of the dislocation loop microstructure in the higher helium experiment but that the degree of refinement is much smaller, on the order of a factor of two or three in the loop density.

In the range 400-500°C the swelling in EBR-II is known to exhibit a minimum transient regime of ~10 dpa prior to swelling at a rate of ~1%/dpa.[3,19] If this trend also pertains to swelling at the lower displacement rate of ORR then these 20-45% Ni alloys would have accumulated 280-580 appm helium prior to reaching the 10 dpa level. The large decrease in swelling at 400°C at all nickel levels relative to that of EBR-II may reflect the impact of the very large and almost unprecedented density of cavities developed prior to the onset of rapid swelling.

The swelling in ORR at 500°C increases with declining nickel content as the void density decreases from the 10^{17} to the 10^{16} cm^{-3} level, but at this temperature in EBR-II it is known that swelling would also increase as the nickel level falls. Thus the behavior observed at 500°C must reflect not only some aspect of the helium-induced refinement but also the earlier observed effect of nickel via its effect on vacancy diffusivity and dislocation bias at low helium levels.[6,20,21]

The cavity sizes induced in ORR at 45% Ni and 500°C, as well as at all nickel levels at 400°C, are small enough that they are most likely helium bubbles rather than voids. At these sizes, the cavities may have been driven below the critical radius for bubble-to-void conversion.[22] Although the swelling in ORR at 400°C is significantly less than that observed in EBR-II, the extensive refinement of the small amount of swelling present is probably the major reason for the much larger level of strengthening observed in the tensile tests conducted on ORR irradiated specimens. Preliminary analysis of the microstructural results indicates that, at all irradiation temperatures studied, the refinement of swelling (along with some refinement of loop microstructure) is sufficient to account for the higher levels of strengthening observed in the ORR experiment.

It should be cautioned, however, that the helium/dpa ratios employed in the ORR test are much larger than anticipated in proposed fusion neutron spectra. As shown in the next section, similar tests conducted at more appropriate helium/dpa levels using isotopic doping do not show such a large effect of helium on microstructure and tensile properties.

Isotopic Tailoring

The most promising approach currently available to explore the separate and synergistic effects of helium and other variables in nickel-containing alloys involves the use of isotopic tailoring of the nickel, which allows side-by-side comparison of helium effects at both low and fusion-relevant generation rates. This technique can be used in a variety of reactor spectra by employing ^{59}Ni additions or by making adjustments in the ^{58}Ni/^{60}Ni ratio.[23]

When ^{59}Ni isotopic doping is combined with the on-line temperature control (±5°C) available in the FFTF Materials Open Test Assembly an excellent opportunity arises to unambiguously separate the contributions of various important variables.[24] Even in the event of nonisothermal or nonisoflux irradiation, the doped and undoped specimens experience exactly the same temperature, spectrum and flux history. For a doped 25% nickel alloy producing 10 appm/dpa in FFTF, the ^{59}Ni contribution contributes only a 0.4% increase in dose rate, producing for the first time a truly one-variable experiment designed to study helium's influence in competition with other important variables. In the experiment described below the higher level of helium/dpa ratio produced with ^{59}Ni was designed to be approximately the same at both 25 and 45% nickel, thereby allowing observation of nickel's effect without introducing another contribution to the helium generation rate.

In the first series of studies employing isotopic doping with ^{59}Ni, the interactive effects of helium, dpa level, temperature, nickel level, phosphorus addition and cold working are being investigated with respect to their impact on microstructure and mechanical properties.[15-17,26] In this experiment, shown schematically in Figure 6, the helium/dpa ratios chosen are more representative of a fusion environment. While the higher levels of helium generation rate were shown to influence somewhat the details of the microstructure (Figure 7), the effect of helium on macroscopic properties was small in general and secondary to that of all other variables studied. Density change data at 495°C are shown as an example in Figure 8. In mechanical property measurements it was clearly shown that in the absence of other perturbing variables, the influence of helium was rather minor, as shown in Figures 9-11. This conclusion is in sharp contrast to the conclusion one could draw in an uncritical assessment of Figure 2. Early results of ongoing tensile tests indicate that this conclusion appears to be valid at other compositions and irradiation temperatures.[26] Further studies now in progress will test the validity of this conclusion at higher fluence and helium levels.

It is interesting to note that the most significant feature of the evolution of yield strength shown in Figures 9-11 is the radiation-induced convergence to a value that encourages further swelling and is dependent on temperature and composition, but not on starting state. This indicates that helium exerts a relatively minor role in the evolution of dislocation and loop microstructure. A similar convergence behavior has been observed in 316 stainless steel irradiated in EBR-II.[27] The evolution of yield strength of stainless steels has been demonstrated to be dependent on displacement rate, however.[28] The introduction of displacement rate as a variable significantly complicates the interpretation of the strength data in Figure 2.

Comparison of the EBR-II and ORR Studies

Helium's influence on microstructural development depends not only on the quantity of helium but its interaction with other variables. While its influence can be observed in the details of the microstructure, its impact on macroscopic property changes in these experiments appears to be relatively small unless helium is generated at rates higher than that anticipated in fusion environments. Comparative experiments directed towards helium's influence on tensile properties but conducted at different displacement rates are more difficult to interpret compared to experiments in which the helium generation rate is the only variable.

THE UNUSUAL BEHAVIOR OF NICKEL

Nickel is often used in fission-fusion correlation efforts directed toward either helium/dpa or PKA recoil spectra effects. Its irradiation behavior is quite different from that of other metals and alloys, however. Operating on the principle that one cannot clearly define the effect of a perturbing variable associated with a simulated environment until the parametric response in the surrogate environment is understood, this author recently reviewed both published and previously unpublished high fluence irradiation data on nickel.[29] It was shown that unlike iron-base FCC alloys, nickel's swelling behavior is dominated not by its tendency to swell initially at a rate comparable to that of Fe-Cr-Ni alloys, but by its persistent tendency to eventually saturate in swelling, a behavior not observed in other metals and alloys.

The saturation level of neutron-induced swelling is usually less than 10%, but the rate of approach to saturation is very sensitive to many environmental and material variables, especially displacement rate, purity and starting dislocation density.[29] At higher irradiation temperatures, helium and other gases also appear to influence the evolution. There appears to be strong interactions between each of these variables.

One aspect of this behavior is the observation that the saturation level of swelling is only weakly dependent on irradiation temperature, especially for those conditions which favor the early establishment of a dislocation network. As a test of this idea, the author recently examined the behavior of 99.995% nickel irradiated in EBR-II. As shown in Figure 12 the swelling of annealed nickel at 14 dpa was a moderately strong function of temperature, but when irradiated in the 30% cold-worked condition, the swelling was relatively insensitive to temperature.

After aging, the swelling actually increased somewhat relative to the cold-worked condition but retained the independence of irradiation temperature. This suggests that annealed specimens experience more difficulty at higher temperatures in generating a stable dislocation network, a problem which is easily overcome in the presence of a preexisting network. The slight increase in swelling with preirradiation aging suggests that there is an optimum initial dislocation density for void swelling that is somewhat lower than that obtained by cold-working. Microscopy examination will be pursued to confirm this hypothesis.

The saturation process appears to be an ubiquitous feature of nickel during irradiation but the approach to and magnitude of the saturation level appears to be very sensitive to variables such as helium, purity and dislocation density. Fission-fusion correlations utilizing nickel should be careful to minimize the number of variables operating in the experiment. Otherwise, apparently insignificant differences in environmental and material variables may overwhelm the effects of helium/dpa ratio or PKA recoil spectra. Whereas

Fe-Cr-Ni alloys develop a saturation microstructure that encourages cavity growth and its associated hardening to continue, nickel develops a saturation microstructure that discourages further swelling. In addition, experiments conducted to dose levels that produce swelling near the saturation level may not be very responsive to changes in other variables such as helium.

An additional complication arises when analyzing experiments involving nickel in reactors which produce a large amount of helium, in that the same transmutation reaction which produces the helium also leads to a significant increase in displacement rate. Greenwood has shown that the displacement rate in pure nickel irradiated in the mixed spectrum reactor HFIR can be increased as much as 90%.[30] This important consideration must be factored into the comparative analysis of irradiations conducted on nickel in different reactor spectra. Unlike copper, solid transmutants are not a significant consideration for irradiations of nickel.

DIVERGENT RESPONSE OF COPPER TO SOLID TRANSMUTANTS

One might suspect that since copper is a relatively soft metal, it might also have trouble establishing a dislocation network and would therefore be subject to saturation of swelling. As shown in Figure 13, however, MARZ grade copper at ~400°C swells at a rate of ~0.5%/dpa with no hint of saturation at 100 dpa.[31] Addition of 5% nickel also does not appear to alter the swelling behavior. This is particularly significant because copper is one material that develops significant amounts of solid transmutants (nickel and zinc). Both of these elements have a pronounced effect on thermal and electrical properties, a most important consideration since copper alloys are candidates for high heat flux service in fusion devices. Nickel's effect on conductivities is much stronger than that of zinc.

Figure 14 shows that the electrical conductivity of copper and some of its alloys indeed falls during irradiation in FFTF at 400°C in response to the combined effect of solid transmutants and swelling. It was earlier shown that these two contributions are predictable and directly additive when no precipitation is involved.[4] The conductivity behavior of copper at 530°C is due almost exclusively to transmutation since the swelling at 33 dpa is only 1.8%, while that at ~400°C is ten times larger. Although it appears that saturation is occurring in conductivity with increasing dpa, the behavior observed is only a consequence of the nonlinearity of copper's response to nickel and zinc as well as the nonlinear dependence of conductivity on voidage.

The amount of solid transmutants is strongly dependent on neutron spectra, with the nickel-zinc ratio increasing in a typical fusion spectrum and thereby causing a larger reduction of conductivity. Thus the data shown in Figure 14 must be modified before application to fusion design, while the data in Figure 13 may be directly applicable.

Figure 14b shows that when small amounts of silver or phosphorus are added to copper, the resultant loss of conductivity is preserved during irradiation to very high dpa levels. There are some types of nonadditive behavior which can occur, however. In a previous publication it was shown that the electrical conductivity of Cu-2.0Be increased slightly with irradiation initially, but remained constant thereafter.[4] The initial increase might be attributed to radiation-induced acceleration of precipitation and aging, but the constant conductivity thereafter was inconsistent with the continued accumulation of nickel and zinc via transmutation.

It now appears that the accumulation of nickel drives some of the beryllium out of solution, and the two effects compensate. The strongly reduced solubility of beryllium in copper containing nickel is the principle on which the higher conducting CuBeNi (Cu-0.3Be-1.8Ni) alloy was developed to replace the lower conductivity alloy Cu-2.0Be.

It also appears that copper alloys may not be very sensitive to helium in their swelling behavior. In recent studies, Zinkle and coworkers employed ^{10}B to study in side-by-side irradiations the effects of ~100 appm helium on microstructure and swelling in pure copper over a wide range of temperature.[32,33] While the details of the microstructure were found to be somewhat sensitive to the ~100 appm helium formed early in the irradiation, the bulk swelling over most of the temperature range of void formation was remarkably similar to that of copper irradiated without boron. To reach this conclusion, however, one must discount the influence of ~100 appm of lithium formed concurrently with the helium.

CONCLUSIONS

Each of the three materials discussed in this paper exhibits a different response at high neutron fluence to factors relevant to the development of fission-fusion correlations. In each case transmutation plays some role in the observed response and in each case the material exhibits a behavior that indicates its role in evolution of properties may overwhelm factors related to PKA recoil spectra. For high fluence experiments, the unavoidable introduction of other variables such as differences in impurities or displacement rate may have a greater impact on the observed behavior than either PKA recoil spectra or helium/dpa ratio.

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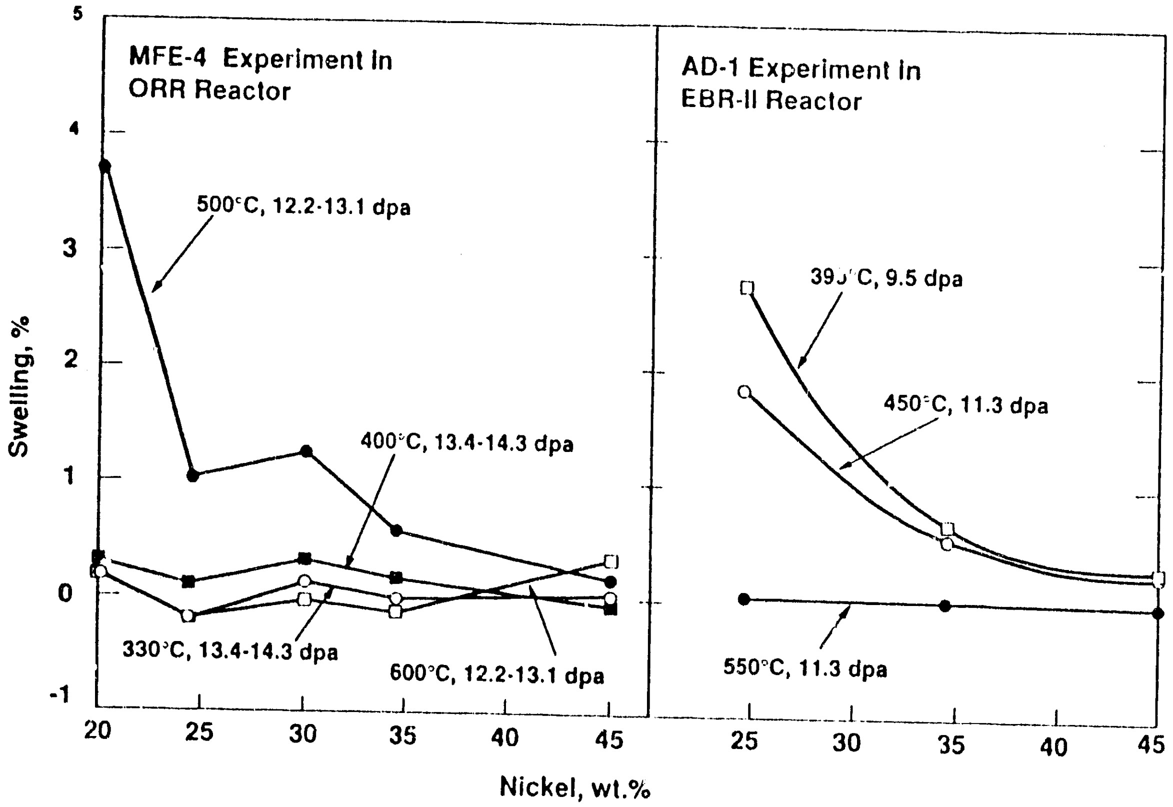


FIGURE 1. Comparison of Swelling Behavior in Fe-15Cr-XNi Alloys in Two Different Reactors[11]

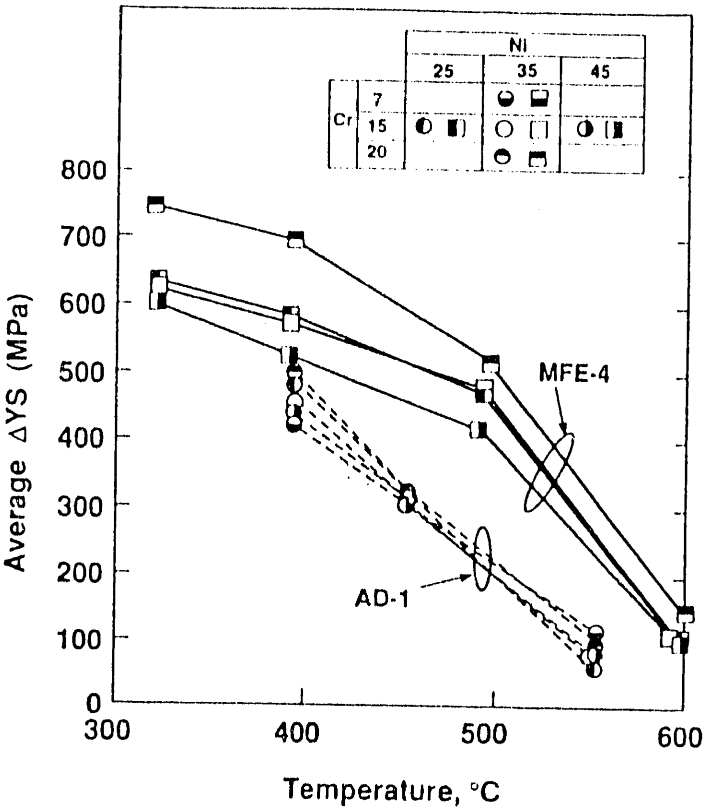


FIGURE 2. Comparison of Radiation-Induced Changes in Yield Strength of Fe-15Cr-XNi and Fe-YCr-35Ni Alloys Irradiated in Either the AD-1 Experiment in EBR-II or the MFE-4 Experiment in ORR[11]

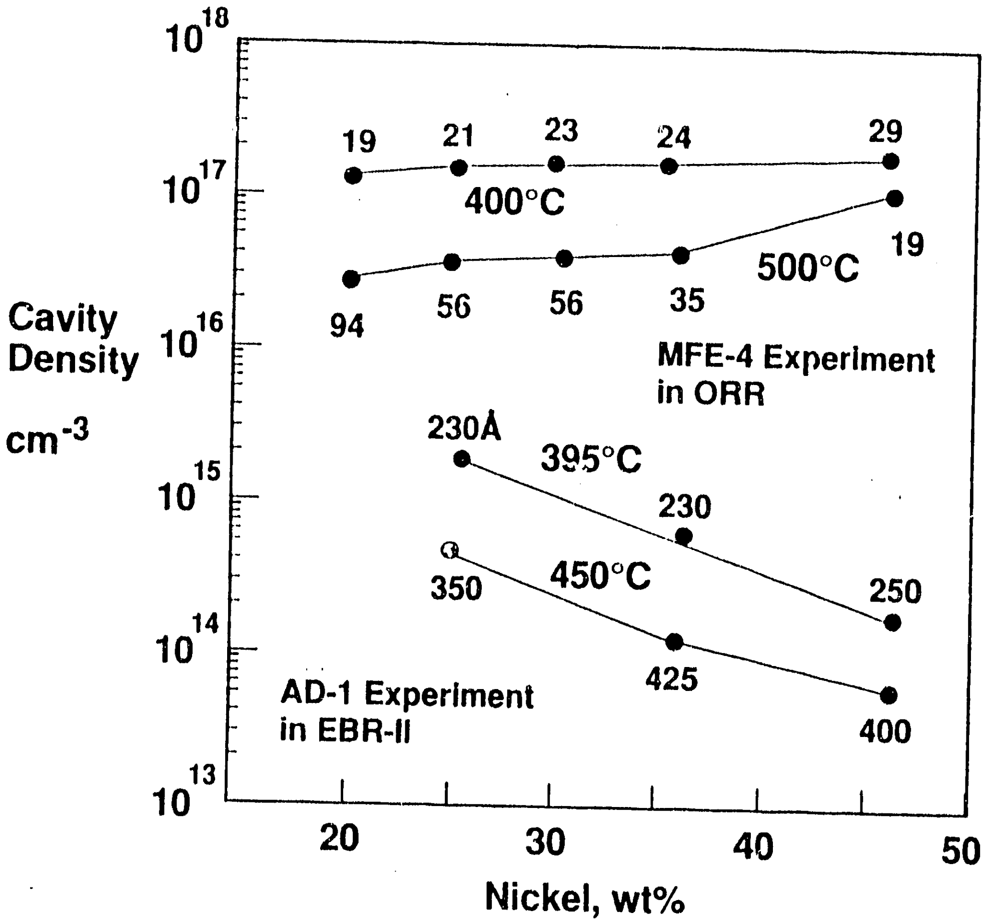


FIGURE 3. Void Densities Observed in the Fe-15Cr-XNi Alloys Irradiated in EBR-II and ORR.[13] Void sizes in Å are also given.

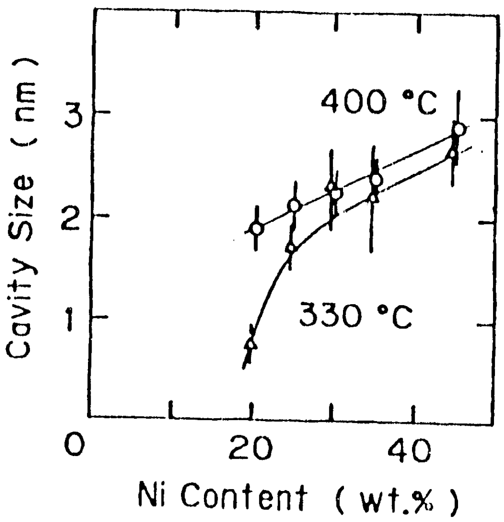


FIGURE 4. Cavity Sizes Observed in Fe-15Cr-XNi Alloys Irradiated in ORR at 330 and 400°C[13]

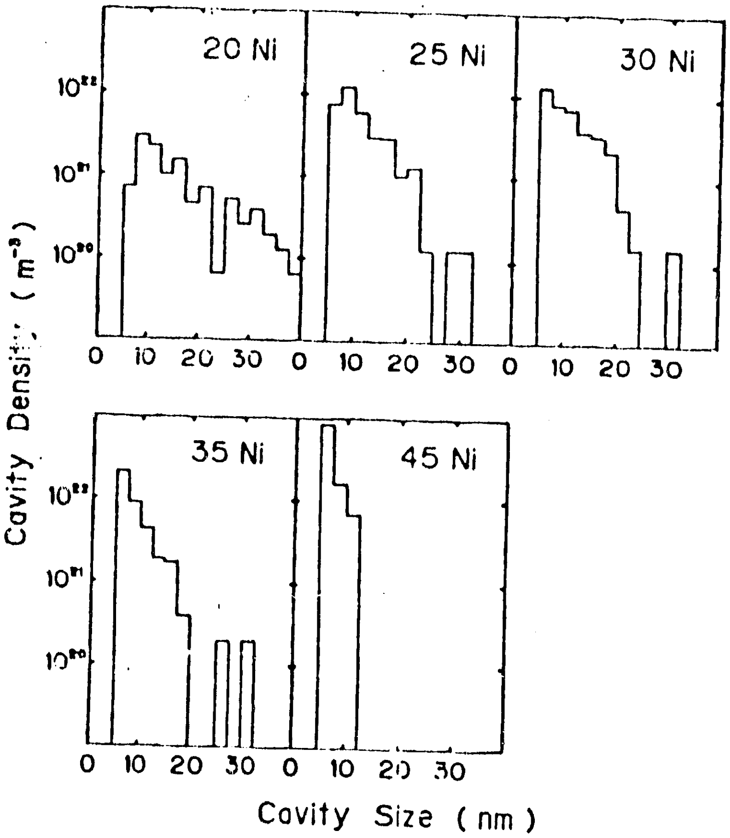
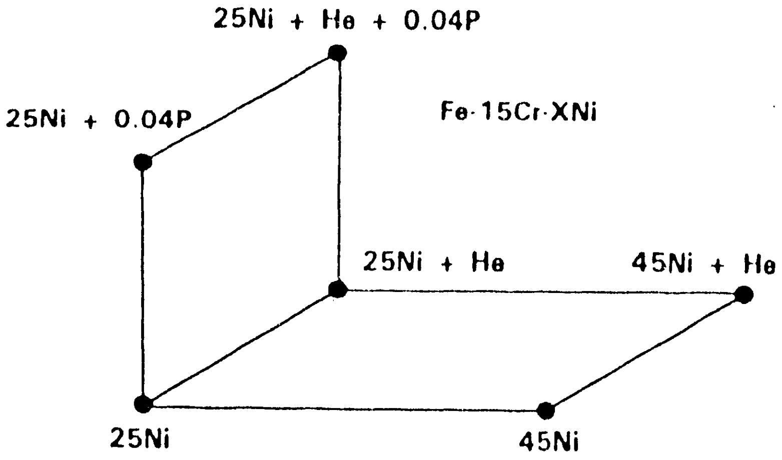


FIGURE 5. Cavity Size Distributions Observed in Fe-15Cr-XNi Alloys Irradiated in ORR at 500°C[13]



- 360, 450, 495, 600°C
- 3 FLUENCE LEVELS
- He/dpa → 4 TO 18 with Ni^{59}
- ANNEALED VS. 20% COLD-WORKED
- NO INFLUENCE OF DISPLACEMENT RATE

FIGURE 6. Schematic Representation of the ^{59}Ni Isotopic Tailoring Experiment

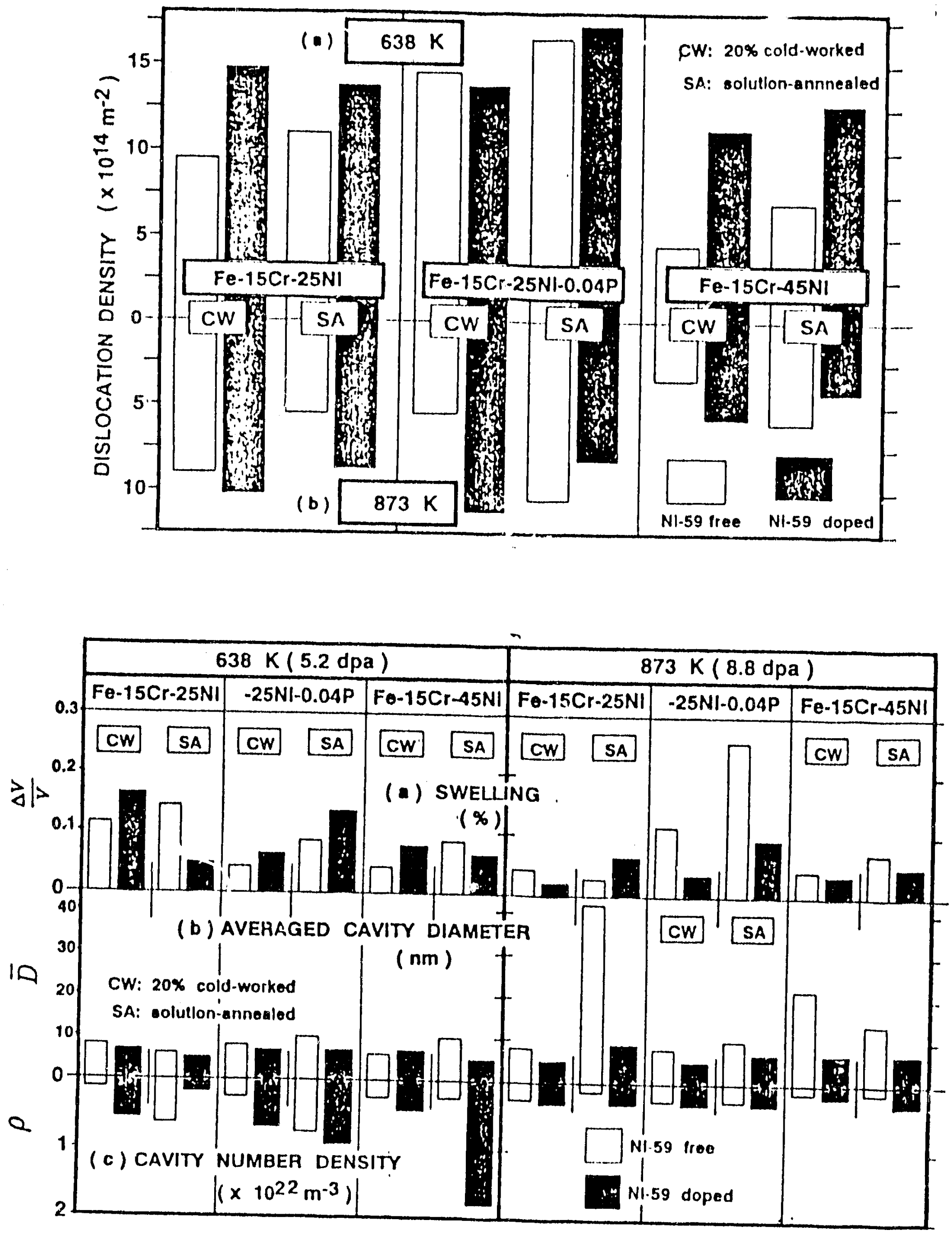


FIGURE 7. Details of Microstructure Observed in Isotopic Doping Experiments at 365°C and 600°C for Helium/dpa Ratios of (0.3 and 13.9) and (0.4 and 4.4) appm/dpa Respectively[17]

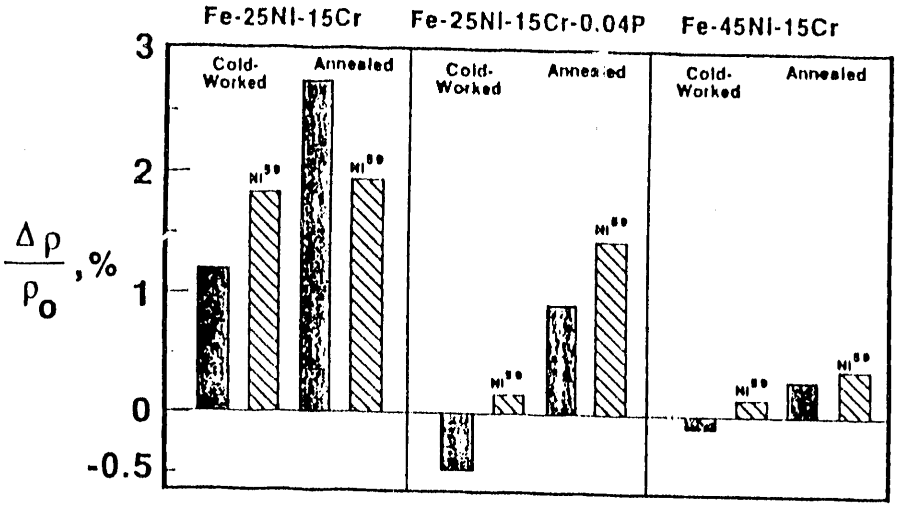


FIGURE 8. Density Changes Observed in the 12 Alloy Conditions Irradiated at 495°C.[16] Helium/dpa ratios are 0.35 and 4.7 appm/dpa for 25Ni and 0.57 and 4.9 appm/dpa for 45Ni.

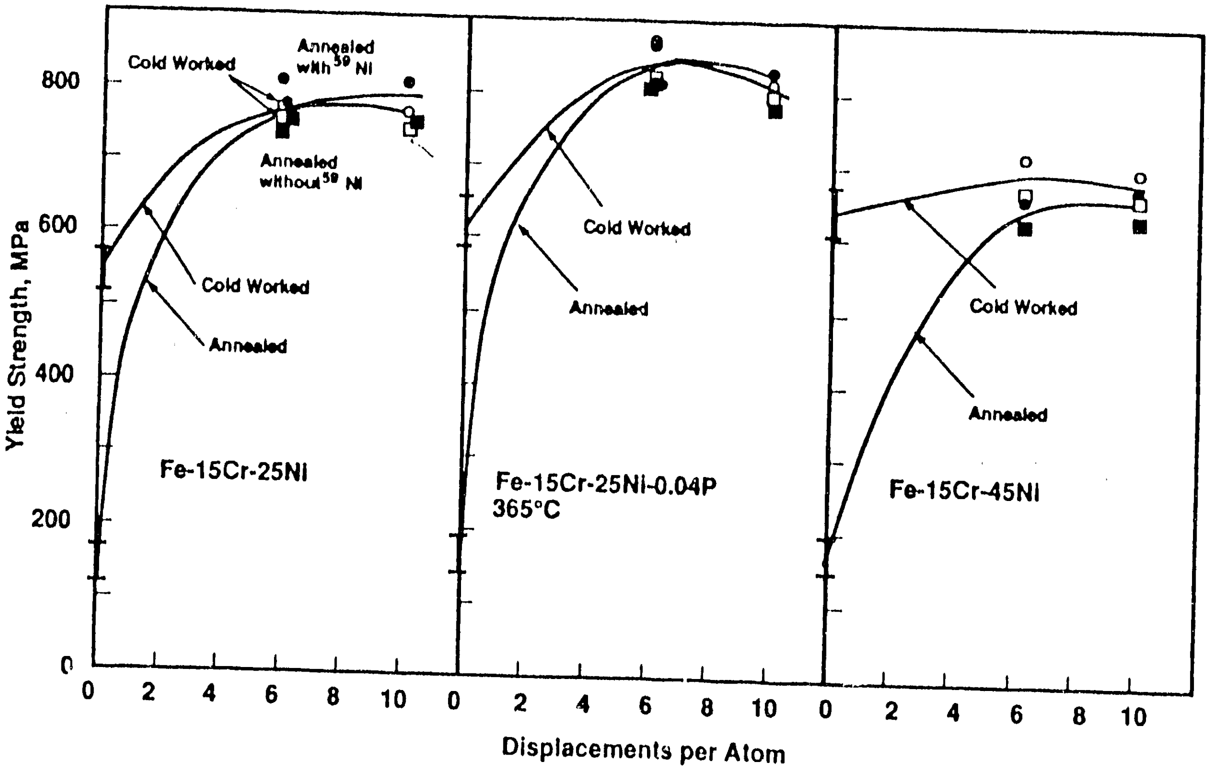


FIGURE 9. Influence of Composition, Thermomechanical Starting State and He/dpa Ratio on Yield Strength Changes of Fe-Cr-Ni Alloys Irradiated at 365°C (He/dpa ratios of 0.3 and 13.9).[26] Open and closed symbols denote cold-worked and annealed alloys, respectively. Circles denote ^{59}Ni-doped specimens; squares denote undoped alloys.

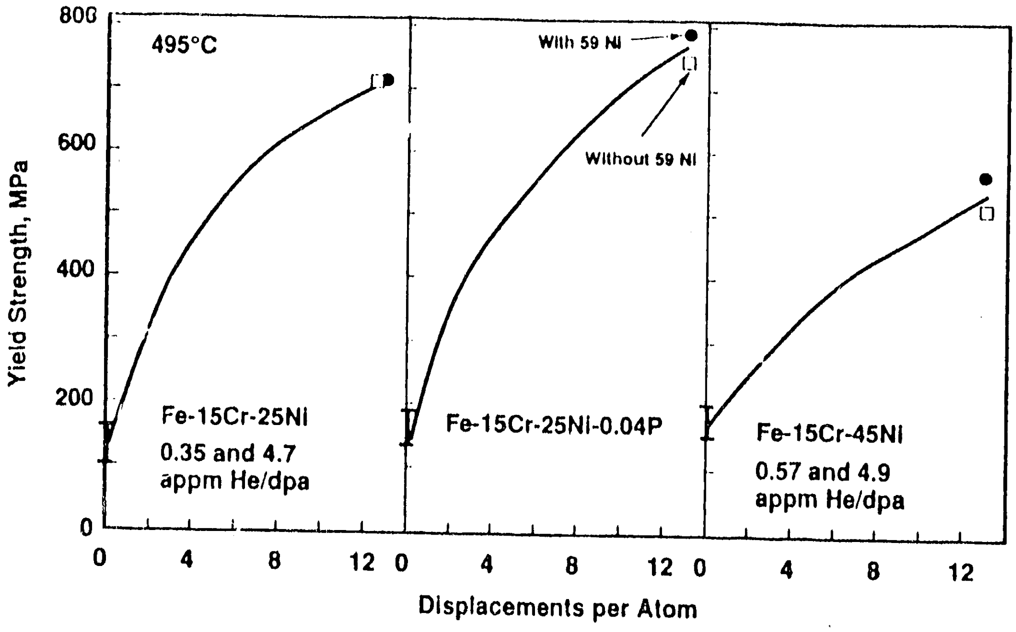


FIGURE 10. Influence of Helium/dpa Ratio on Yield Strength Changes of Irradiated Annealed Fe-Cr-Ni Alloys at 495°C and 13 dpa.[26] The helium/dpa ratios for the undoped and doped alloys both depend somewhat on nickel content.

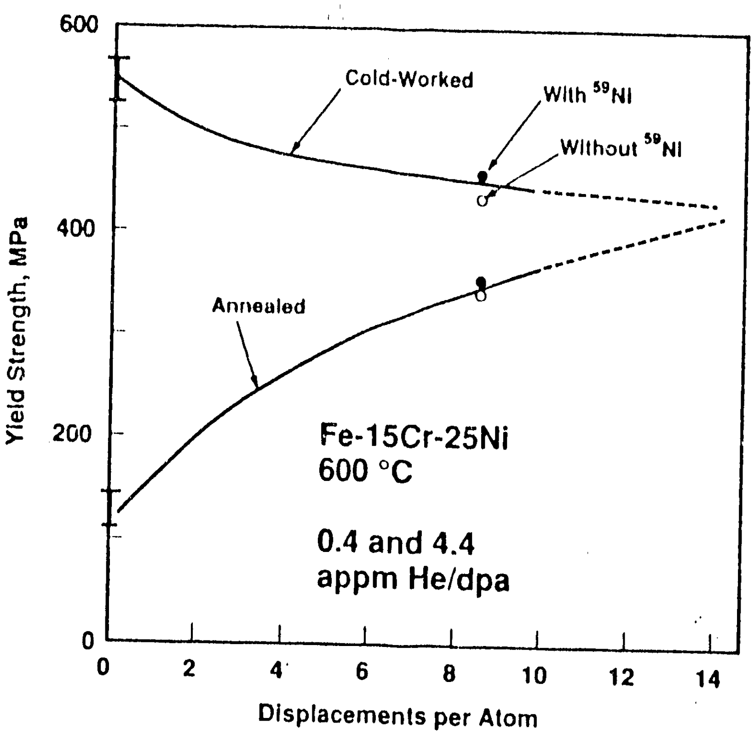


FIGURE 11. Influence of Thermomechanical Starting State and He/dpa Ratio on Yield Strength Changes of Irradiated Fe-15Cr-25Ni at 600°C and 8.7 dpa[26]

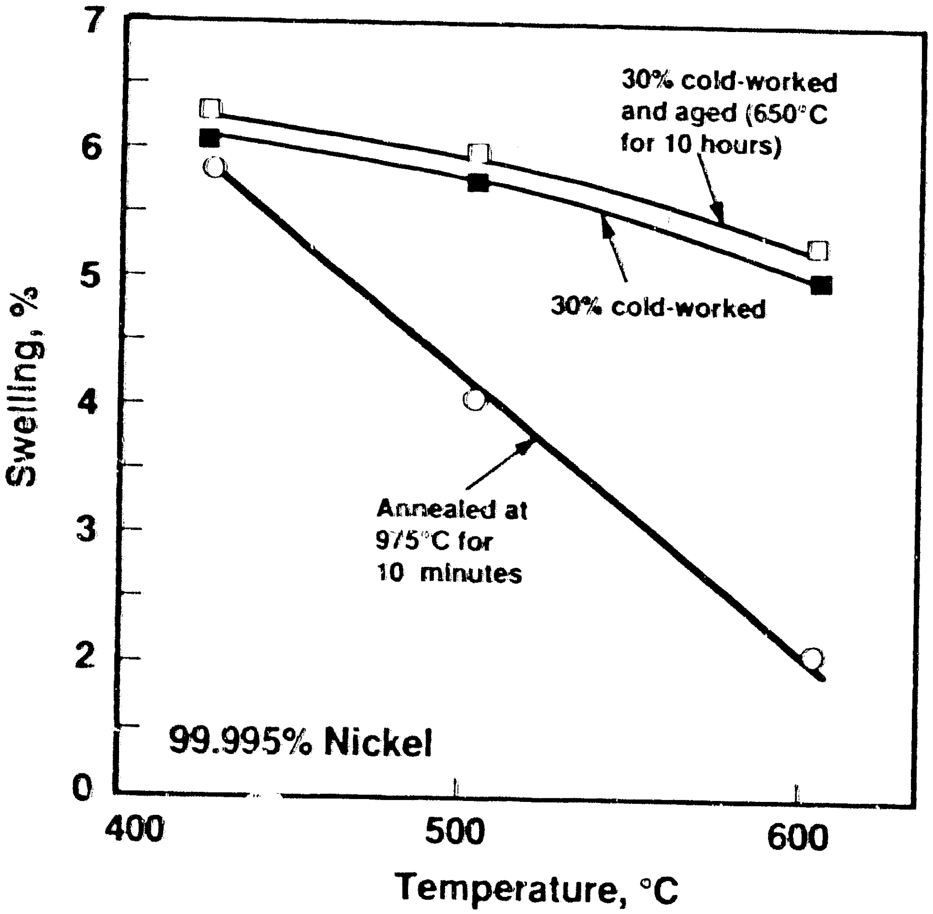


FIGURE 12. Swelling of Nickel Irradiated to 14 dpa in the EBR-II Fusion Materials Experiment Designated AA-14. Three irradiation temperatures and three starting conditions were employed.

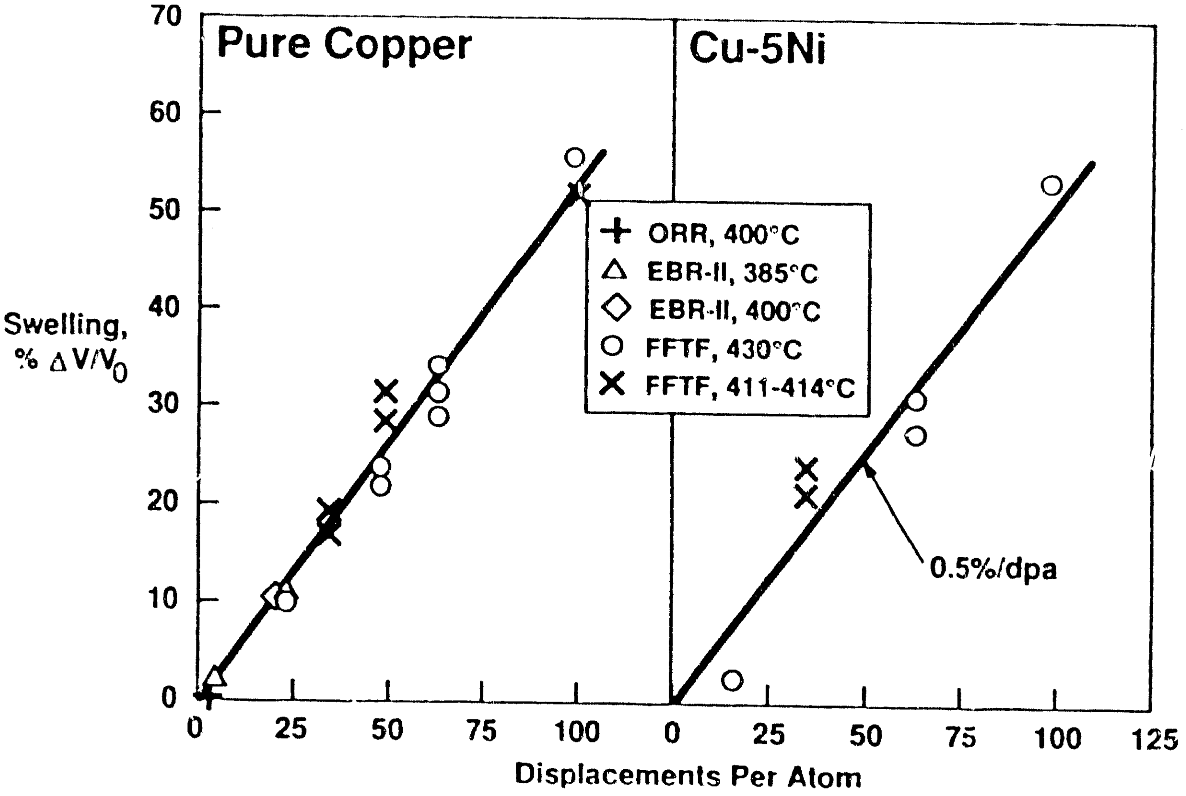


FIGURE 13. Comparison of Swelling Behavior at ~400°C of Pure Copper and Cu-5 wt% Ni in Various Irradiation Experiments.[31]

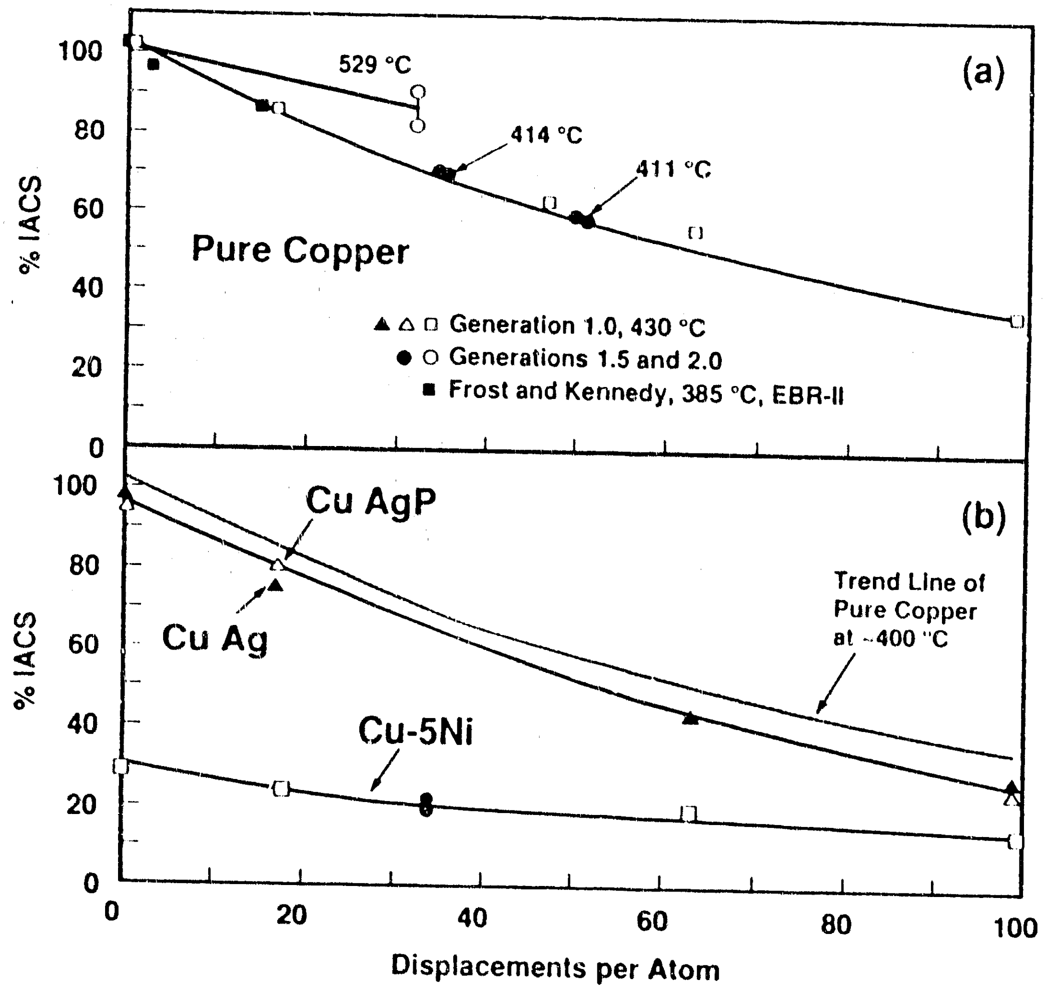


FIGURE 14. Neutron-Induced Changes in Electrical Conductivity of Pure Copper and Some of its Alloys in Various Irradiation Experiments[34]

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