PNL-SA--18854

DE91 004894

DEC 1 4 1990

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November 1990

To be included in Fusion
Materials Semiannual Progress
Report DOE/ER-0313/9

Work supported by
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

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THE INFLUENCE OF NICKEL CONTENT ON MICROSTRUCTURES OF Fe-Cr-Ni AUSTENITIC ALLOYS IRRADIATED WITH NICKEL IONS

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OBJECTIVE

The objective of this effort is to identify the mechanisms involved in the radiation-induced evolution of microstructure in materials intended for fusion applications. The results of this study are useful in interpreting the results of several other ongoing experiments involving either spectral or isotopic tailoring to study the effects of helium on microstructural evolution.

SUMMARY

Ion-irradiated Fe-15Cr-XNi (X = 20, 35, 45, 60, 75) ternary alloys and a 15Cr-85Ni binary alloy were examined after bombardment at 675°C and compared to earlier observations made on these same alloys after irradiation in EBR-II at 510 or 538°C. The response of the ion-irradiated microstructures to nickel content appears to be very consistent with that of neutron irradiation even though there are four orders of magnitude difference in displacement rate and over 200°C difference in temperature. It appears that the transition to higher rates of swelling during both types of irradiation is related to the operation of some mechanism that is not directly associated with void nucleation.

PROGRESS AND STATUS

Introduction

In an earlier report, the microstructures were examined for a series of Fe-15Cr-XNi (X = 15, 19, 21, 30, 35, 45, 75) ternary alloys and a 15Cr-85Ni binary alloy after irradiation in the EBR-II reactor at 510 and 538°C.[1] At 510°C it was obvious that void nucleation was complete prior to the onset of steady-state swelling at all nickel levels, implying that some process other than void nucleation determines when the relatively rapid void growth associated with steady-state swelling occurs. At both 510 and 538°C a protracted loop dominance at intermediate nickel levels was observed in the dislocation microstructure along with a relatively low void density, suggesting a reduced bias toward preferential interstitial absorption as a possible mechanism to produce lower swelling in this nickel range. The void densities also closely mirrored the total swelling behavior as a function of nickel content. Figures 1-4 show the relationships observed between loop and void densities, swelling, dislocation density and matrix nickel content.

It was decided to test the generality of such observations over a broad range of irradiation conditions. The first results of some other neutron irradiation studies are presented elsewhere.[2] A more rigorous test of the generality of these microstructural relationships was provided by microscopy observations of a series of specimens which were ion bombarded at much higher displacement rates, then thinned and observed by Johnston, Rosolowski, Turkalo and Lauritzen.[3] These specimens were found to be in excellent condition more than 15 years after their original examination.

Experimental Details

The materials examined in this study were Fe-15Cr-XNi (X = 20, 35, 45, 60, 75) ternary alloys and a 15Cr-85Ni binary alloy. These alloys were prepared separately by Johnston and coworkers and thus are nominally but not fully identical to those used in the neutron studies. The irradiations were performed with either 4 or 5 MeV Ni^+ ions at 675°C reaching 30 to 190 dpa at a displacement rate of about 2×10^{-2} dpa/s. Prior to irradiation, 20 appm of helium had been preinjected to depths where the microscopy was to be performed. The regions of the specimen examined by TEM were 700 to 850 nm from the ion-incident surface of the specimen. Due to depth-dependent changes in displacement rate characteristic of heavy ion bombardment, variations in depth of the foil from the incident surface resulted in variations in total dose. The alloys and irradiation conditions examined are listed in Table 1. The microstructure and microchemistry of these specimens were examined with a JEM-2000FX TEM equipped with an EDS system.

RESULTS AND DISCUSSION

Since ion milling was performed by Johnston and coworkers at the final stage of specimen preparation, high densities of small dots were observed in all specimens. Stereoscopic observations were carried out to distinguish between heavy ion-induced microstructure inside the specimen and near-surface defect clusters induced by ion milling. Micrographs of the damage structures will be presented in another report.[5]

(a) Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

Table 1

Alloys (Fe-15Cr-XNi or 15Cr-85Ni) and Heavy Ion Irradiation Conditions. The irradiation temperature is 675°C.

Ni level (wt%)	20	35	35	35	45	45	45	60	75	85
Ni^+ ion energy (MeV)	5	4	4	5	4	5	4	5	5	5
Dose	30	30	53	110	58	120	190	111	116	115

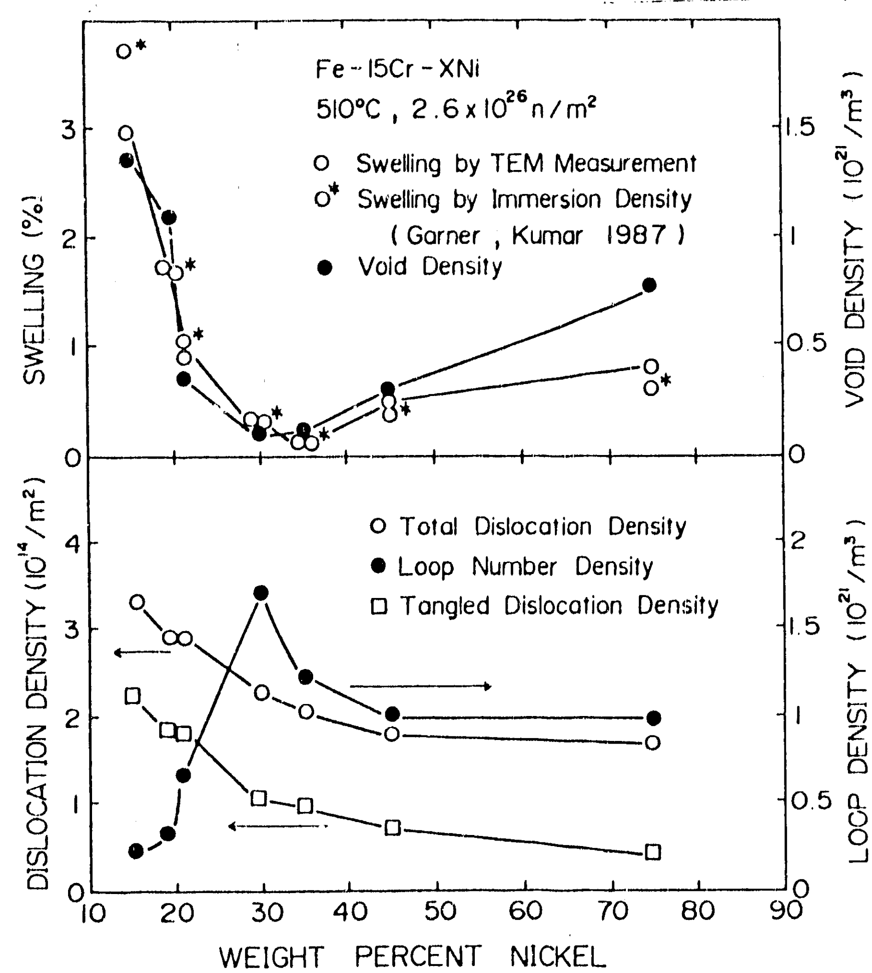

Figure 1. Dependence of microstructural parameters on nickel content at 510°C and 2.6×10^{26} n/m^2 (E > 0.1 MeV).[1]

Figure 5 shows the measured microstructural parameters as a function of nickel content. Although only lower dose microstructures were available for 20Ni and 35Ni alloys, the results concerning the nickel dependence are qualitatively similar to those shown in Figures 1 and 2 except that both void density and swelling for the 85Ni binary alloy are higher than those of the 75Ni ternary alloy. Garner[6] has shown that irradiation of 85Ni-15Cr with either ions or neutrons yields different results, depending on whether the irradiation is performed above or below 550°C, the order-disorder transition temperature for this alloy. Thus one would expect some difference in behavior for neutron irradiations conducted at 510°C or 538°C and ion irradiations conducted at 675°C.

Figure 6 shows the dose dependence of both swelling and void density for the 35 and 45Ni ternary alloys. The swelling under the accelerated irradiation conditions of ion bombardment at higher temperature indeed

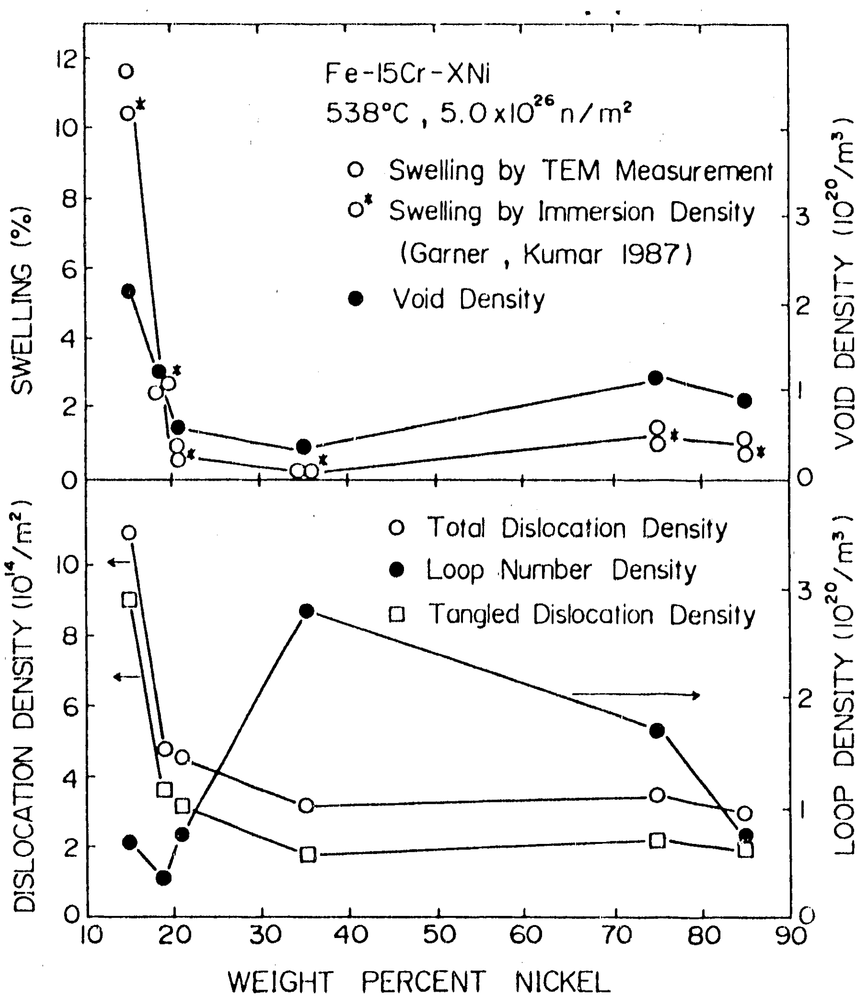

Figure 2. Dependence of microstructural parameters on nickel content at 538°C and 2.6 x 10^{26} n/m^2 (E > 0.1 MeV).[1] Swelling data obtained by immersion density by Garner and Kumar[4] are also shown.

appears to obey the kinetics of transient and post-transient rapid increase observed in neutron irradiation at lower displacement rate and temperature. Figure 6 also shows that the change in void density is small during the transition between these two regimes, similar to the behavior of the neutron-irradiated specimens shown in Figure 3.

Profiles of nickel concentration across voids of about 100 nm in diameter are presented in Figure 7 for a variety of nickel levels. The enrichment of nickel at voids is most prominent for lower nickel alloys. Figure 8 shows, for the 45Ni ternary alloy, the dose dependence of swelling as well as the dose dependence of nickel concentration at void surfaces and in the matrix. The nickel depletion in the matrix coincides with the onset of rapid swelling, once again consistent with the behavior of the neutron irradiated 30Ni ternary alloy shown in Figure 4.

CONCLUSION

The nickel-dependent microstructural response of Fe-Cr-Ni alloys irradiated under very different conditions appears to be quite consistent. It appears that void densities tend to saturate at nickel dependent levels rather early in the irradiation. The transition to higher rates of swelling seems to require the later operation of some other mechanism not directly associated with void nucleation.

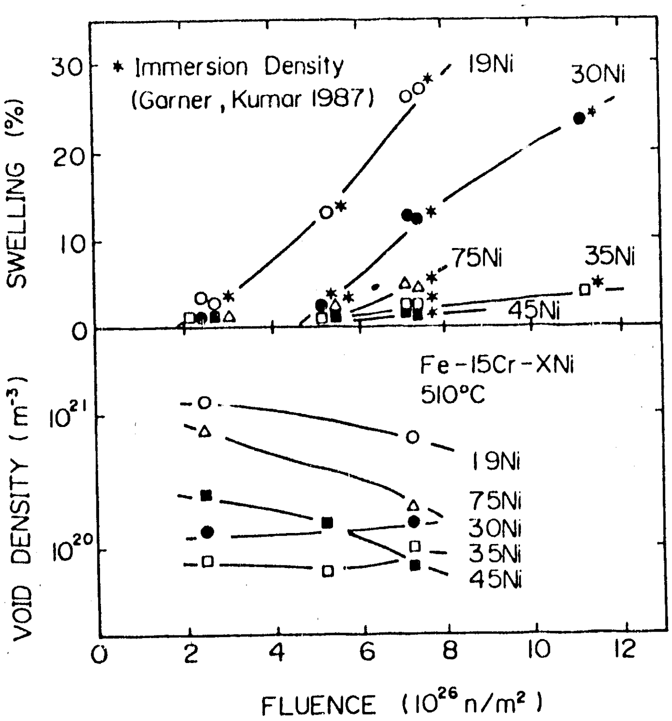

Figure 3. Fluence dependence of swelling and void density at 510°C.[1] Swelling data obtained by immersion density by Garner and Kumar[4] are also shown.

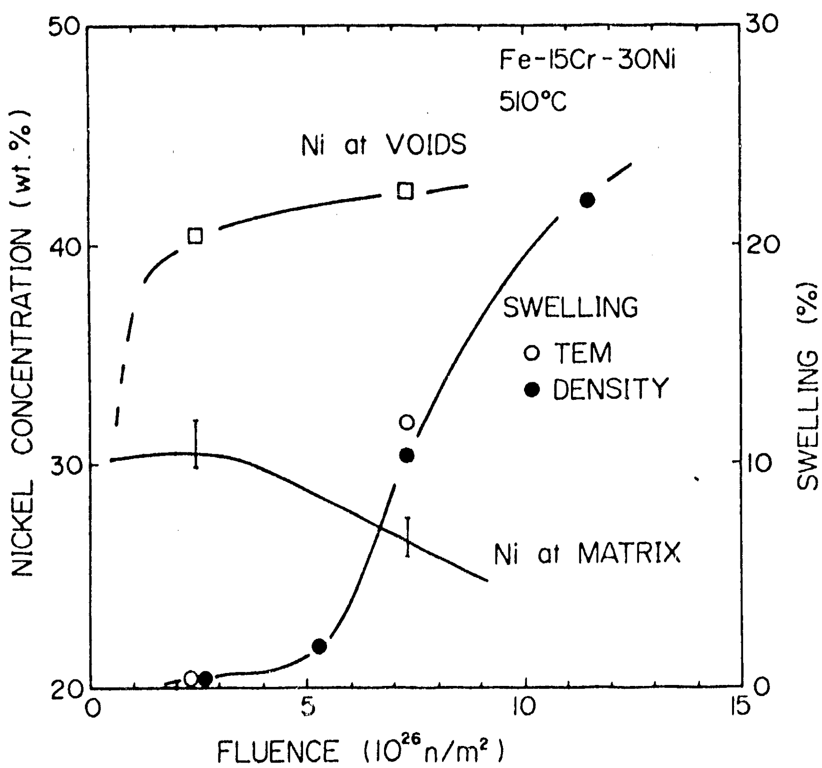

Figure 4. The fluence dependence of swelling and nickel concentration at void surfaces and in the matrix of Fe-15Cr-30Ni irradiated at 510°C[1]

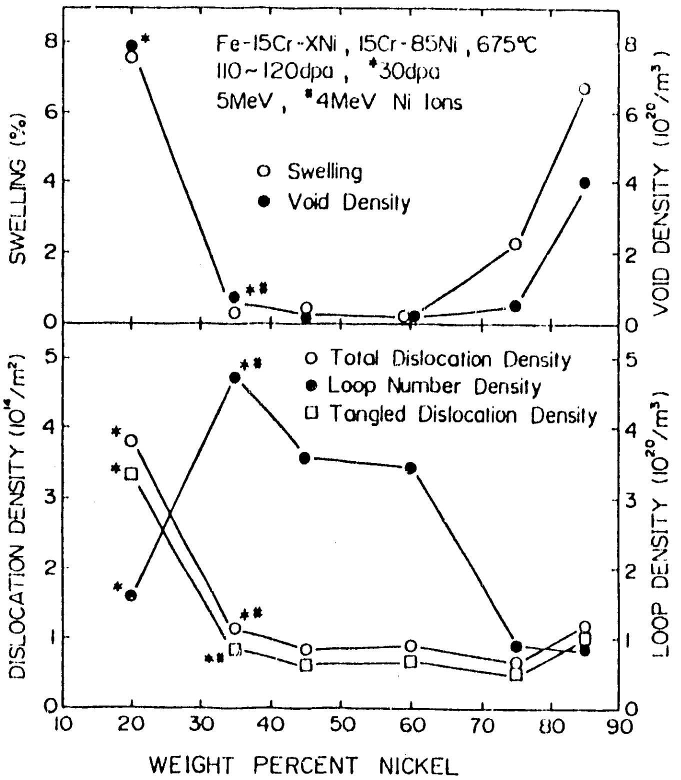

Figure 5. Dependence of microstructural parameters on nickel content at 675°C and either 30 or 111-120 dpa after irradiation with either 5 or 4 MeV Ni ions.

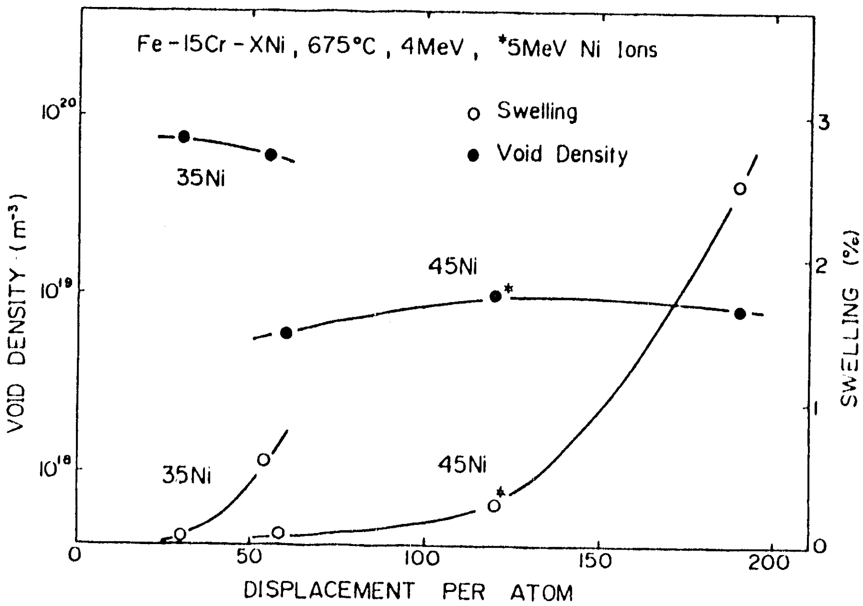

Figure 6. Dose dependence of swelling and void density in Fe-15Cr-XNi alloys irradiated with either 4 or 5 MeV Ni ions at 675°C

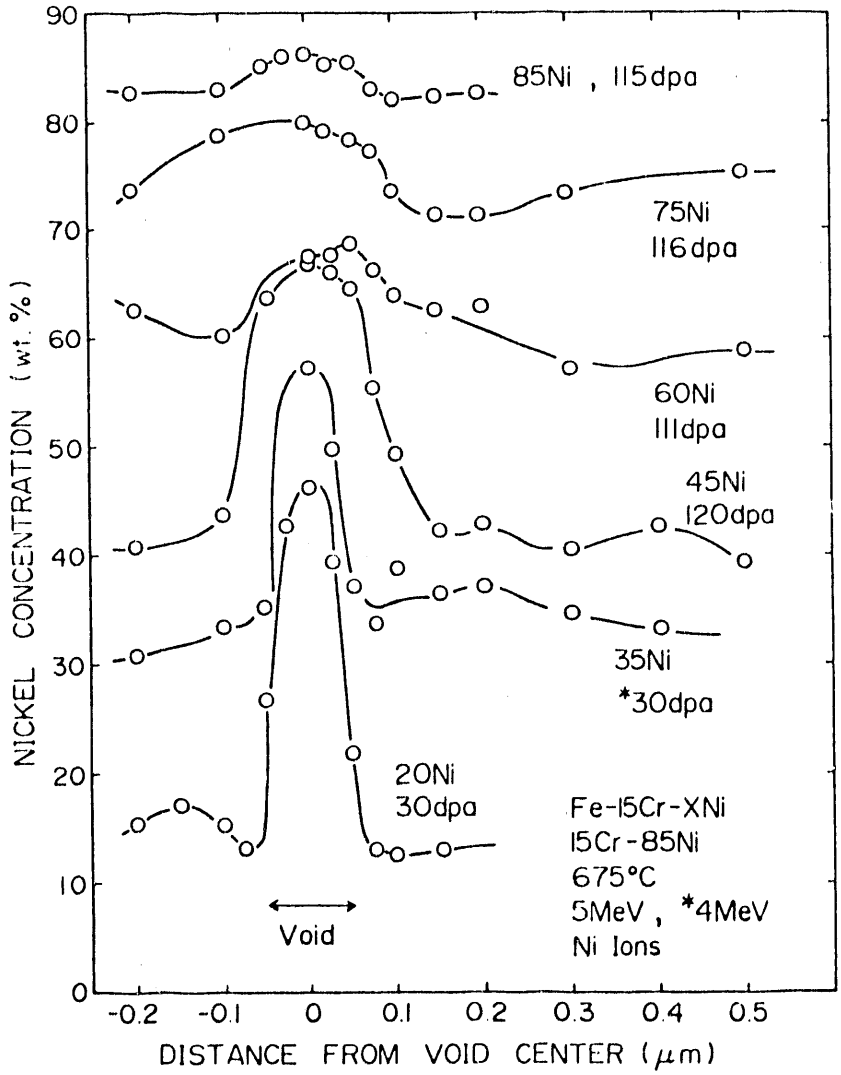

Figure 7. Profiles of nickel concentration across areas containing voids of about 100 nm in diameter for alloys irradiated with either 5 or 4 MeV Ni ions at 675°C.

FUTURE WORK

These results will be combined with the early neutron results and some theoretical analysis. These will then be published in the proceedings of the 15th ASTM Symposium on Effects of Radiation on Materials.[5]

ACKNOWLEDGMENTS

This work was supported in part by the Japan-USA Fusion Cooperation in "Fundamental Studies of Irradiation Effects in Fusion Materials Utilizing Fission Reactors" sponsored by Monbusho, the Japanese Ministry of Education, Science and Culture. The work was also supported by the U.S. Department of Energy under Contract DE-AC06-76RLO 1830.

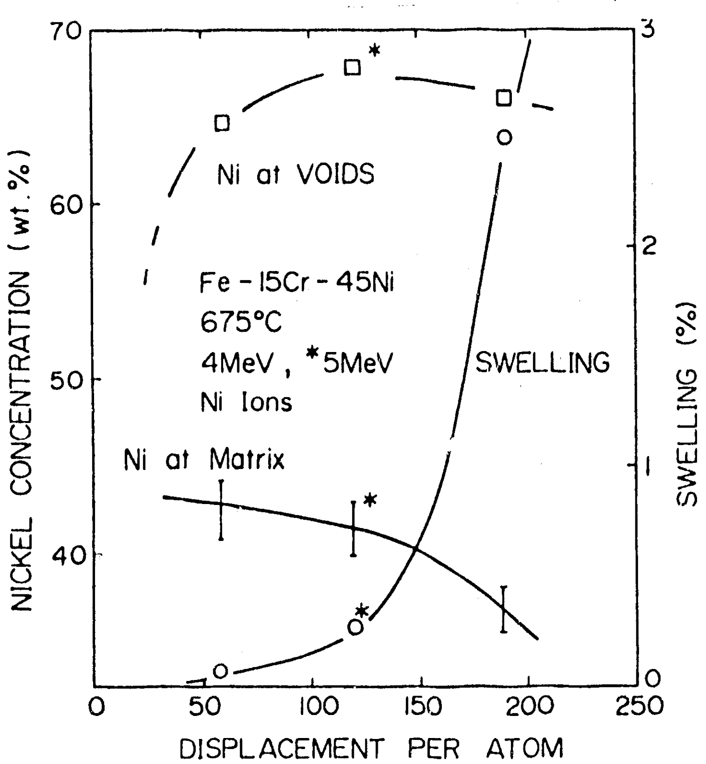

Figure 8. The dose dependence of swelling and nickel concentration at void surfaces and in the matrix of Fe-15Cr-45Ni irradiated with 4 or 5 MeV Ni ions at 510°C

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