

PNL-SA--19346

Re

DE92 004505

THE INFLUENCE OF HELIUM ON MECHANICAL  
PROPERTIES OF MODEL AUSTENITIC ALLOYS  
DETERMINED USING <sup>59</sup>Ni ISOTOPIC TAILORING  
AND FAST REACTOR IRRADIATION

M. L. Hamilton  
F. A. Garner

November 1991

Presented at the  
5th International Conference on  
Fusion Reactor Materials  
November 17-22, 1991  
Clearwater, Florida

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

Pacific Northwest Laboratory  
Richland, Washington 99352  
**DISCLAIMER**

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

**MASTER**

**DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED**

*1/10/92*

THE INFLUENCE OF HELIUM ON MECHANICAL PROPERTIES OF MODEL  
AUSTENITIC ALLOYS, DETERMINED USING  $^{59}\text{Ni}$  ISOTOPIC TAILORING AND  
FAST REACTOR IRRADIATION

M. L. Hamilton and F. A. Garner  
Pacific Northwest Laboratory  
Richland, WA

ABSTRACT

Tensile testing on model Fe-Cr-Ni alloys removed from four discharges of the  $^{59}\text{Ni}$  isotopic doping experiment in FFTF indicates that helium/dpa ratios typical of fusion reactors do not produce changes in the yield strength or elongation that are significantly different from those at much lower helium generation rates. It also appears that tensile properties approach a saturation level that is dependent only on the final irradiation temperature, but not prior temperature history or thermomechanical starting condition.

1. Introduction

Until recently it has been impossible to conduct experiments in which spectrum-related parameters such as helium/dpa ratio were varied without also accepting variations in other important parameters such as displacement rate or temperature history. A technique currently being used, however, allows the study of the influence of helium alone on density change, microstructural evolution and mechanical properties. This technique utilizes

isotopic tailoring to vary the helium production rate without introducing changes in neutron spectrum or displacement rate.<sup>1,2</sup> It is possible to generate substantial variations in He/dpa ratio without varying any other important parameter by using alloys between which the only difference is the presence or absence of <sup>59</sup>Ni, an isotope that does not occur naturally, and irradiating doped and undoped specimens side by side in the appropriate reactor spectra.<sup>1</sup>

A particular advantage of comparative isotopic doping experiments is that one need not be concerned with the details of temperature history, which are now known to heavily influence the outcome of some fission-fusion correlation experiments.<sup>3</sup> Since both doped and undoped specimens are irradiated side by side, the primary variable is only the helium/dpa ratio. The production rate of helium in doped specimens is also nearly, but not exactly, constant throughout the experiment, providing that no changes occur in the neutron environment. Small variations in helium production rate occur in both sets of alloys in response to burn-in or burn-out of <sup>59</sup>Ni.<sup>4</sup>

This paper presents data obtained from miniature tensile specimens irradiated in an experiment conducted in the Fast Flux Test Facility (FFTF) utilizing the Materials Open Test Assembly (MOTA).

A previous paper presented the results of the first discharge of this experiment.<sup>5</sup> The details of the helium measurements are included in another paper.<sup>4</sup>

## 2. Experimental Details

The alloys employed in this study were nominally Fe-15Cr-25Ni, Fe-15Cr-25Ni-0.04P and Fe-15Cr-45Ni (wt%) in both the cold worked and annealed conditions. These alloys were chosen to complement those used in several earlier studies, one in the Experimental Breeder Reactor-II (EBR-II), designated the AD-1 experiment,<sup>6,7</sup> and another conducted in the Oak Ridge Research Reactor, designated MFE-4.<sup>7</sup> The acquisition of the <sup>59</sup>Ni, the production of the <sup>59</sup>Ni-doped tensile specimens, and their irradiation conditions are described elsewhere.<sup>1</sup> Microscopy disks were also prepared and irradiated; the results of transmission electron microscopy (TEM) examination are described in detail in References 8-10. The miniature tensile specimens nominally measured 5.1, 1.0 and 0.25 mm in gauge length, width and thickness, respectively. They were tested at room temperature at a strain rate of  $4.7 \times 10^{-4} \text{ sec}^{-1}$  in a horizontal test frame described in Reference 11. Yield strengths were determined at 0.2% offset. More than one tensile specimen was tested for some conditions, but the majority of test conditions involved only a single specimen.

Table 1 shows the discharges from four of the irradiation sequences of the  $^{59}\text{Ni}$  experiment, each defined by its target irradiation temperature and its location in FFTF/MOTA. The experiment was initiated in MOTA 1D, but a short temperature excursion referred to as an overtemperature event compromised the integrity of parts of the MOTA. A decision was made to run the MOTA in the helium-purged mode for the remainder of FFTF cycles 7 and 8 while a series of reactivity feedback tests were conducted. The majority of the MOTA canisters therefore operated at variable but lower temperatures until the end of MOTA 1D.

Table 1. Irradiation Sequence for  $^{59}\text{Ni}$  Experiment

Irradiation Temperature (°C)	Relative Location	Accumulated Neutron Damage, dpa			
		MOTA 1D	MOTA 1E	MOTA 1F	MOTA 1G
365	Below core	6.1	10.2	--->	~24
495	Level 1	13.9	27.9	--->	~52
"	Level 1	-	14.0	~29	~39
600	Level 5	8.7	-	-	-
490	Above core	2.0	-	-	-
"	Above core	-	2.1	4.8	~6

Two of the compromised experimental sequences were restarted with duplicate specimens in MOTA 1E for isothermal irradiations at the same temperatures, 490 and 495°C. The original 490 and 600°C sequences were not continued to higher radiation exposure due to

the magnitude of the temperature excursion, during which these canisters experienced 553 and 806°C, respectively, for fifty minutes. The 495°C sequence also experienced an overtemperature, reaching 629°C for fifty minutes, but was continued in the irradiation sequence along with the replacement sequence at 495°C to allow an evaluation of temperature history effects. The sequence at 365°C did not experience a temperature excursion during MOTA ID and continued irradiation isothermally as planned.

### 3. Results of Tensile Tests

It was shown in earlier papers that the doped and undoped specimens exhibited the same tensile properties in the unirradiated condition<sup>5</sup> and that there was excellent agreement between the data from the miniature specimens used in this experiment and the data from two types of larger specimens employed in other experiments.<sup>7</sup> Figures 1 through 4 show the yield strength and elongation data obtained at all four irradiation temperatures. The helium generation rates given are averages over the first two irradiation segments. Helium measurements have not yet been completed for the last segment.

The width of the error bars at zero dpa corresponds to the variability in the data found in the earlier study<sup>5</sup> and provides a

basis for determining whether variations observed between doped and undoped specimens are significant compared to the scatter associated with the measurement technique. The most significant feature of the data shown in Figures 1 through 4 is the relative unimportance of isotopic doping at all test temperatures in determining the yield strength. Also significant is the tendency toward convergence of the data on annealed and cold worked specimens to saturation levels that depend only on alloy composition and irradiation temperature. A similar convergence was observed previously in 316 stainless steel over a wide range of irradiation temperatures, with convergence levels sensitive to both temperature and displacement rate.<sup>12,13</sup>

At 495°C only annealed tensile specimens were irradiated and it is therefore not possible to confirm that convergence occurs at this temperature. TEM disks were irradiated at 495°C in both the annealed and cold worked conditions, however, and convergent microstructures appear to have developed.<sup>8,10</sup> Convergence is clearly occurring in the 490°C sequence, however, conducted at a lower displacement rate and a higher helium generation rate.

The data at 495°C demonstrate another type of convergence. Note that at 495°C the strength of the original specimens (i.e., those that were subjected to the overtemperature and subsequent low

temperature irradiation) initially reached a very high level and then fell to lower levels during the second and third irradiation sequences, while the specimens in the replacement sequence reached the same lower levels directly. Similar but inverted behavior is evident in the elongation data at 495°C, reaching lower ductility levels initially, followed by higher ductility levels when the originally intended irradiation temperature was reestablished. Thus the microstructure and tensile properties converge at a level dependent more on the final irradiation temperature than on earlier temperature history.

The high strength levels that were reached originally did not arise from the temperature excursion itself, however, but from the prolonged irradiation at lower temperatures that followed the overtemperature event. The higher density of microstructure and the resulting higher strength and lower ductility that developed at the lower temperatures were then replaced by microstructures and tensile properties more appropriate to the temperatures achieved in the second and third irradiation segments.

It appears that the helium generation rate in this experiment is of minimal importance compared to the other variables studied. In the absence of variations in displacement rate, the influence of helium is minor. A similar conclusion was reached by Mansur and



Grossbeck<sup>14</sup> in a comparison of data from EBR-II and the High Flux Isotope Reactor on the Japanese and U.S. versions of PCA, the Prime Candidate Alloy of the fusion materials program. Although the displacement rates in the two reactors are comparable, the differences in helium generation rates in that comparison were even larger than in the current experiment.

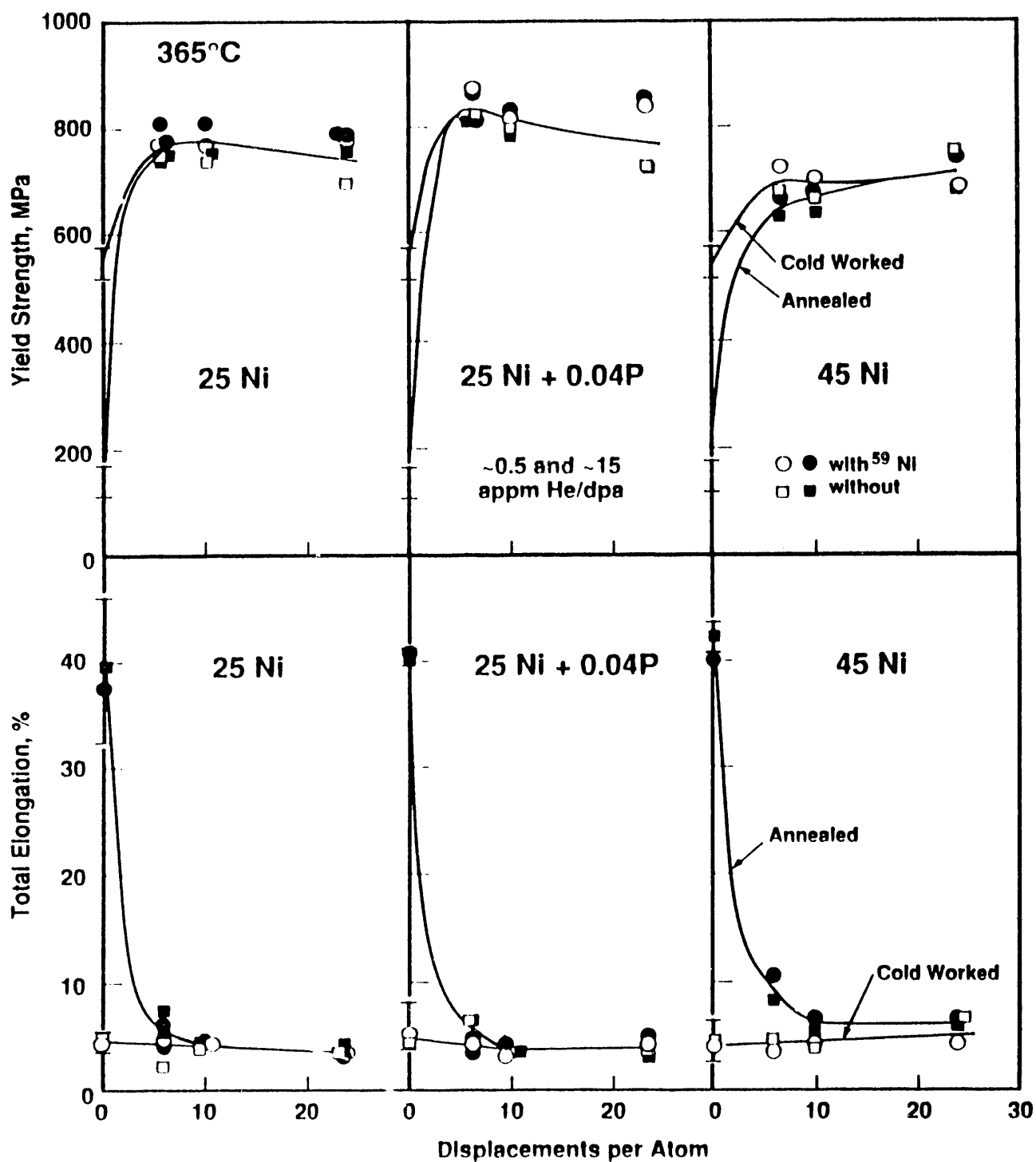
#### 4. Conclusions

When model Fe-Cr-Ni alloys are irradiated at constant displacement rate but at very different helium/dpa rates in the range 365 to 600°C, there is no significant variation in their tensile properties as a function of helium. The impact of helium generation rate is secondary to the influence of cold work, temperature, nickel level and phosphorus level. It appears that tensile properties converge to a saturation level that is dependent on the composition and recent irradiation temperature but not on early temperature history or helium generation rate.

#### 5. References

1. R. L. Simons, H. R. Brager and W. Y. Matsumoto, J. Nucl. Mater. 141-143 (1986) 1057-1060.
2. G. R. Odette, J. Nucl. Mater. 141-143 (1986) 1011-1017.
3. M. Kiritani, J. Nucl. Mater. 160 (1988) 135-141.
4. L. R. Greenwood, F. A. Garner and B. M. Oliver, this conference.

5. F. A. Garner, M. L. Hamilton, R. L. Simons and M. K. Maxon, J. Nucl. Mater. 179-181 (1991) 554-557.
6. H. R. Brager, F. A. Garner and M. L. Hamilton, J. Nucl. Mater. 133 & 134 (1985) 594-598.
7. M. L. Hamilton, A. Okada and F. A. Garner, J. Nucl. Mater. 179-181 (1991) 558-562.
8. J. F. Stubbins and F. A. Garner, J. Nucl. Mater. 179-181 (1991) 573-525.
9. H. Kawanishi, F. A. Garner and R. L. Simons, J. Nucl. Mater. 179-181 (1991) 511-514.
10. J. F. Stubbins and F. A. Garner, this conference.
11. N. F. Panayotou, S. D. Atkin, R. J. Puigh and B. A. Chin, The Use of Small-Scale Specimens for Testing Irradiated Material, ASTM STP 888, W. R. Corwin and G. E. Lucas, Eds., American Society for Testing and Materials, Philadelphia, 1986, pp. 201-219.
12. F. A. Garner, M. L. Hamilton, N. F. Panayotou and G. D. Johnson, J. Nucl. Mater. 103 & 104 (1981) 803-808.
13. F. A. Garner, H. L. Heinisch, R. L. Simons and F. M. Mann, Radiation Effects and Defects in Solids 113 (1990) 229-255.
14. L. K. Mansur and M. L. Grossbeck, J. Nucl. Mater. 155-157 (1988) 130-147.



39111050.30

Figure 1. Influence of thermomechanical starting state and isotopic doping on yield strength and elongation of three alloys irradiated below the core at 365°C.

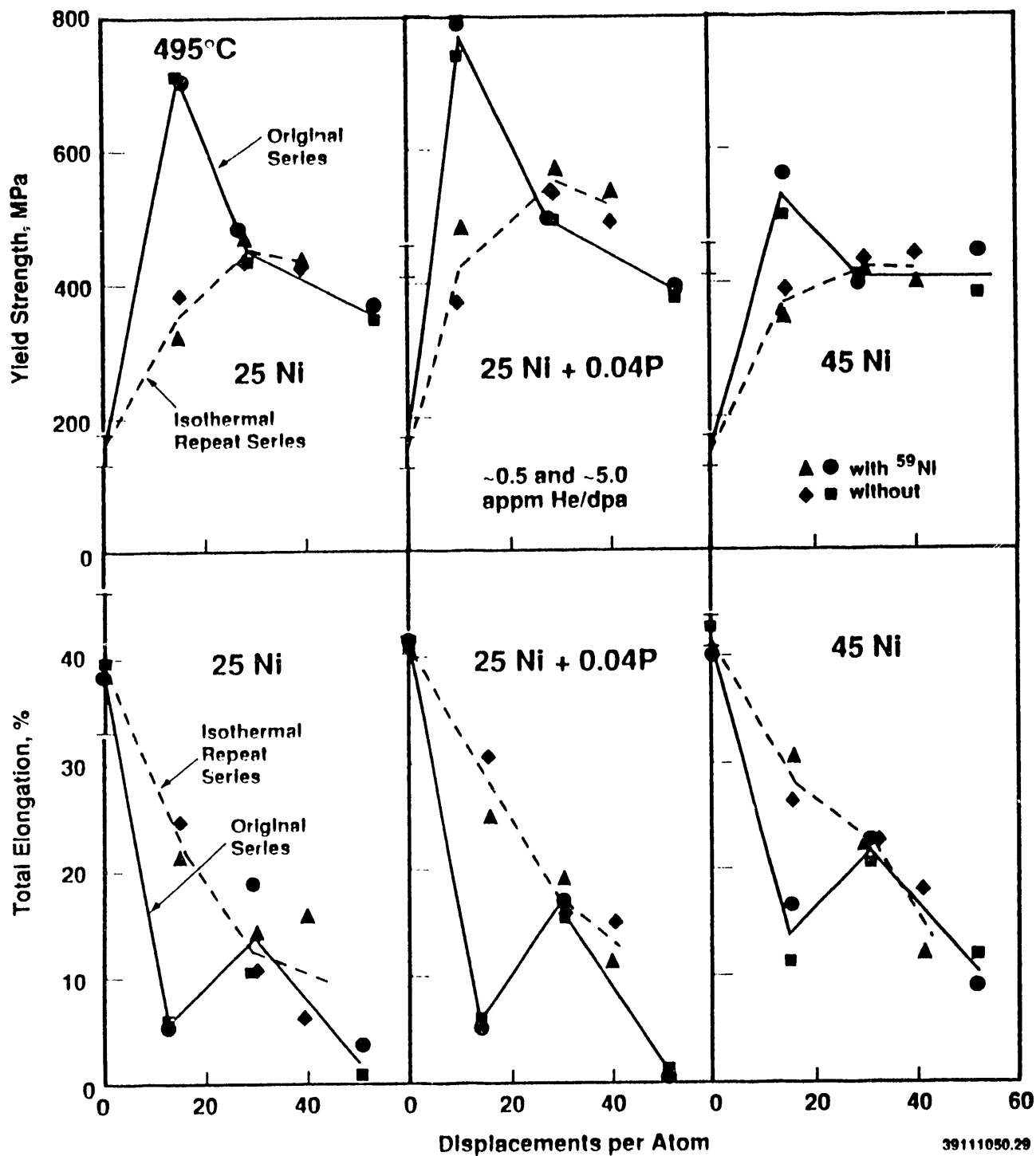
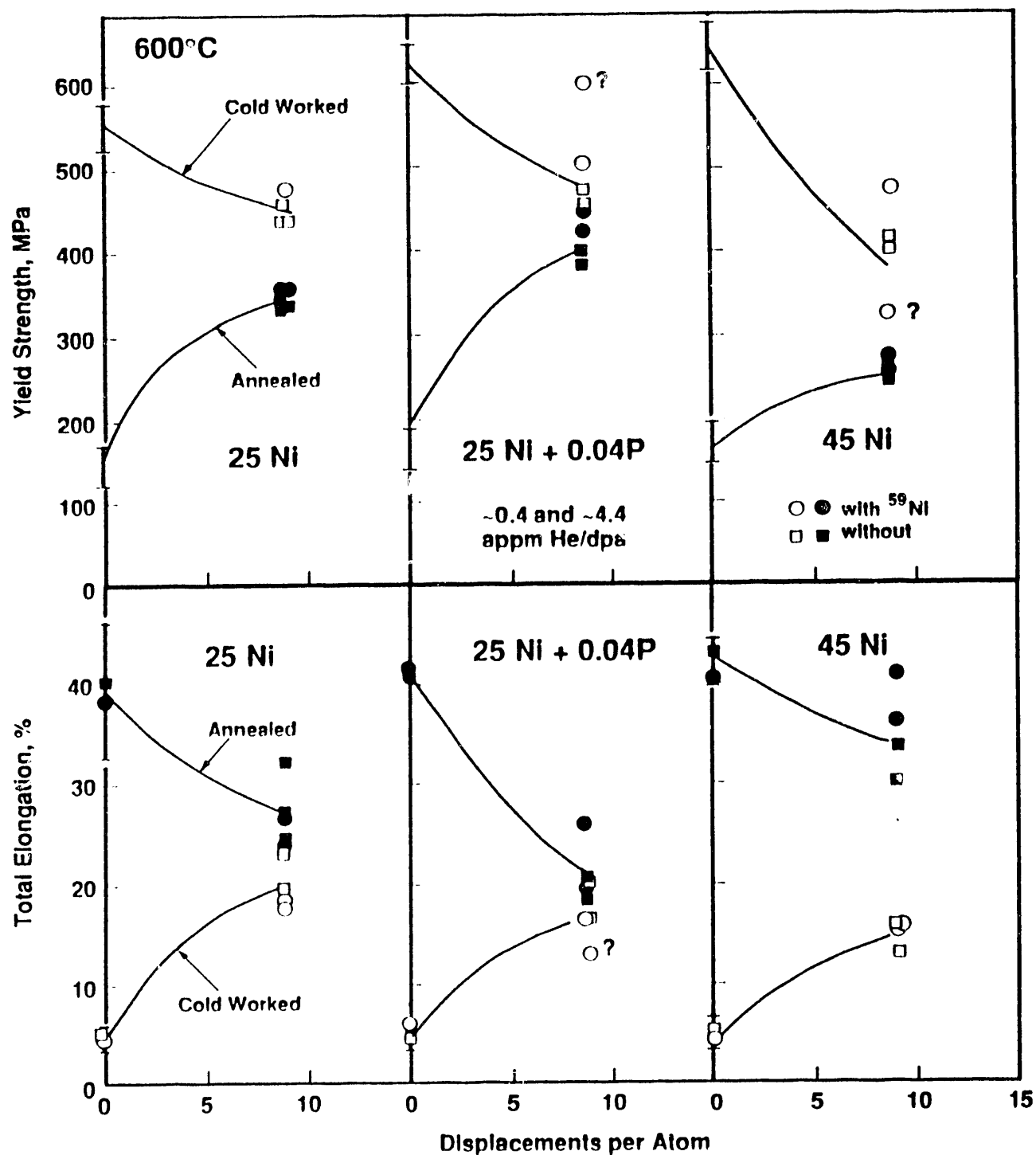


Figure 2. Influence of isotopic doping and temperature history on the yield strength and elongation of annealed alloys following irradiation at the bottom of the core at 495°C. The dotted line corresponds to the isothermal repeat sequence.



39111050.32

Figure 3. Influence of isotopic doping and thermomechanical starting state on yield strength and elongation following irradiation at the top of the core at 600°C.



**Figure 4. Influence of thermomechanical starting state, isotopic doping and temperature history on yield strength and elongation following irradiation above the core at 490°C. The dotted line corresponds to the isothermal repeat sequence.**

**END**

**DATE  
FILMED**

*2 / 20 / 92*

