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Fusion Technology Institute
University of Wisconsin
1500 Engineering Drive
Madison, WI 53706

http://fti.neep.wisc.edu

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J.J. Kai
G.L. Kulcinski
R.A. Dodd

Fusion Technology Institute
University of Wisconsin
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J.J. Kai, G.L. Kulcinski and R.A. Dodd

Fusion Technology Institute, Nuclear Engineering Department
University of Wisconsin-Madison, 1500 Johnson Drive
Madison, WI 53706-1687 USA

Abstract

A set of HT-9 specimens uniformly pre-implanted with 100 appm of helium up to a depth of 1.3 μm were irradiated with 14 MeV nickel ions up to 60 dpa at temperatures of 400, 500 and 600°C. Cavities were observed in the He-doped specimens irradiated at 500 and 600°C. The maximum local swelling was found to be 0.1% in the specimen irradiated at 500°C to 60 dpa. There was no measurable difference in precipitate evolution and dislocation loop structure between samples irradiated with and without pre-implanted helium.

It is concluded that free gas atoms are essential to the formation of cavities in heavy ion irradiated HT-9. The overall low swelling characteristics of this alloy show that HT-9 is a highly swelling resistant material even in the presence of considerable amounts of helium.

1. Introduction

Ferritic and martensitic steels are considered to be the leading candidates for the cladding and structural materials of fast breeder reactors and the first walls and blankets in conceptual fusion reactor designs [1]. Among this class of alloys, the HT-9 ferritic steel has received considerable attention because, in addition to its superior resistance to void swelling [2-8], it also appears to satisfy other reactor requirements such as in-reactor creep resistance [9,10], elevated temperature mechanical properties [11,12], and resistance to helium embrittlement [13].

The present study is a part of a larger effort devoted to the investigation of the microstructural stability of the HT-9 alloy following heavy ion irradiation. The results of microstructural evolution of HT-9 following Ni ion irradiation without helium implantation will be reported in detail elsewhere [14]. This study focusses on the effects of pre-implanting helium into the HT-9 alloy followed by Ni ion irradiation in order to investigate the effects of helium on the evolving microstructures.

2. Experimental Procedures

The microstructure of the unirradiated steel used in this study has been discussed previously [15]. The chemical composition (in weight percent) of this alloy is shown in Table 1.

Foil specimens with dimensions of 5 x 10 x 0.76 mm were mechanically polished and finished with 0.3 μm alumina powder to a smooth surface. Prior to Ni ion irradiation, these foils were evenly pre-implanted with 100 appm helium in a zone extending from the surface to a depth of about 1.3 μm. This pre-implantation is accomplished by injecting He^+ ions with energies varying from 200 to 700 keV (in 100 keV increments) at room temperature. These specimens were then irradiated at 400, 500, and 600°C to total dose levels of 3×10^{20}, 8×10^{20}, and 15×10^{20} ions/m^2 (the highest dose only at 500°C). These doses are equivalent to about 10 dpa, 30 dpa, and 60 dpa, respectively, at the 1.3 μm depth (the maximum depth of helium pre-implantation) and about 40 dpa, 100 dpa, and 200 dpa, respectively, at the peak damage region (which contained no helium). The curves of the calculated dpa and ion-deposition distribution versus depth for 14 Mev Ni ions injected into HT-9 are shown in Fig. 1. The shaded area in Fig. 1 represents the region that was pre-implanted with 100 appm helium. The irradiation temperatures are accurate to ±5°C.

Preparation of the specimens for post-irradiation transmission electron microscopy (TEM) analysis involved the cross-section technique which has been described elsewhere [16]. This procedure allows the entire damage region (see Fig. 1) to be examined in a single TEM specimen. The electron microscopy was performed using a JEOL 200CX TEMSCAN electron microscope operating at 200 kV.

3. Results

One effect of pre-implanting helium into HT-9 specimens followed by ion irradiation is the formation of gas-filled cavities or bubbles in the helium pre-implanted region. Figure 2 shows helium bubbles, which were observed in the specimen irradiated at 600°C to 10 dpa, in under-focus, exact-focus, and over-focus conditions. Figure 3 shows voids observed in the specimens irradiated at 500°C to dose levels of 30 dpa and 60 dpa, and which presumably were nucleated at helium bubble sites. In the 30 dpa specimen, voids were heterogeneously distributed in various regions. The number density is very low ($\leq 1 \times 10^{17}$ m^{-3}) and the total swelling is negligible. In the 60 dpa specimen, voids were also heterogeneously distributed in various regions although the number density and average diameter were larger. The total swelling, however, was still low. Table 2 summarizes the quantitative data of cavities observed in this study. Figure 4 illustrates the swelling versus dose level relationship. It is noticed that even the highest local swelling rate is less than 0.01 %/dpa.

Helium pre-implantation resulted in very little effect on other microstructural features, dislocation loops and precipitates. Figure 5 shows the TEM microstructures in the helium pre-implanted region of the HT-9 specimens irradiated to 10 dpa and 30 dpa. At 400°C the major feature of the entire damage region is a high number density of small dislocation loops, and there is virtually no difference between the microstructure of specimens irradiated with or without helium under otherwise identical conditions. Cavities were not observed in the specimens irradiated at 400°C.

At 500°C, the microstructure of the specimen irradiated to 10 dpa showed no observable voids or new precipitates and the microstructure was the same as that found in the specimen which did not contain helium. However, in the specimen irradiated to 30 dpa, voids were formed (see Fig. 3 (a) and (b)) and some of the voids existed simultaneously with a high number density of small helium bubbles (see Fig. 6 (a) and (b)). In addition to the voids, the microstructure also contained a radiation-induced new phase and a few large dislocation loops and segments. The new precipitate phase was identified as chi phase [17]. Figure 7 shows the chi-phase precipitates in the specimens irradiated at 500°C to peak damage levels of 40, 100, and 200 dpa, respectively. At 600°C the only distinguishable difference between irradiated and unirradiated regions is that small helium bubbles are found in the helium pre-implanted region. It is noted that most bubbles were formed along the subgrain boundaries and the interfaces between precipitates and matrix (see Fig. 2 and Fig. 6 (c) and (d)).

The critical cavity size for void nucleation in HT-9 following heavy ion irradiation can be roughly determined by using the procedures similar to those reported by Horton and Mansur [30]. Following their procedures, the critical cavity diameter of HT-9 irradiated at 500°C is greater or equal to 2.5 nm and at 600°C it is greater than 5.2 n .

4. Discussion

The major effect of pre-implanting helium into HT-9 followed by heavy ion irradiation is the formation of cavities in the helium pre-implanted region. At damage levels as low as 10 dpa, helium bubbles were observed in the specimen irradiated at 600°C. A small amount of void swelling was observed in the specimens irradiated at 500°C to dose levels of 30 dpa and 60 dpa. In contrast, there were virtually no cavities at all in the same alloy following only ion irradiation up to 200 dpa [14]. It was also found in other experimental studies that cavities can be formed in HT-9 during ion irradiation to > 25 dpa with implantation of helium [6] or with as little as 1 appm helium pre-implantation [7].

These results indicate that free gas atoms in HT-9 play a major role in void formation and growth. Farrell [18] reviewed the helium effects on void swelling and concluded that helium is the most important inert gas in nuclear structural materials and is primarily active as a void nucleant and has no direct effects on void growth. Wolfer and Garner [19] also pointed out that helium affects mainly the incubation dose for swelling but not the final rate of steady-state swelling.

The overall low swelling of HT-9 alloy found in this study is consistent with the results of other experimental studies of ferritic steels [2-8,20]. Several mechanisms have been presented to explain the low swelling behavior exhibited by ferritic alloys [21-25]. Sniegowski and Wolfer [21] suggested that the net bias difference between fcc and bcc structures initiated the inherent difference of the swelling resistance between austenitic steels and ferritic steels. Their study showed an order of magnitude difference in steady state swelling rate between austenitic steels and ferritic steels (1 %/dpa vs. 0.1 %/dpa). Little [22] suggested that the existence of strong point defect trapping with solutes will increase recombination rates, which together with dislocation-solute interactions decreasing the bias of dislocations for interstitials, will result in low swelling in ferritic steels. Hayns and Williams [23] presented a model based on incorporating point defect trapping into a rate theory model and found low swelling in ferritic alloys. Little and co-workers [3,24] also presented another explanation involving two types of dislocation loops with Burgers vectors of a <100> and a/2 <111> with different bias formed during irradiation which might result in suppression of void nucleation. Other explanations include the effect of fine grain size on void formation [25], and the initial high dislocation density and precipitate density in the matrix which increases the point defect recombination rate, resulting in lower void swelling.

The low swelling rate found in this study is also consistent with the results of other experiments [7,26,27]. The 0.003 %/dpa (minimum) local swelling rate shows the superior swelling resistance of this alloy. It is noted, however, that the dose level (60 dpa in this study) probably is not sufficient to establish the steady state swelling rate [28] and that it is possible that values higher than 0.003 %/dpa may occur.

The present results indicate that an excess of void nucleation sites is formed in the specimen irradiated at 500°C to 30 dpa with 100 appm pre-implanted He, and it also suggests that cold pre-implantation of He produces many more nucleation sites than produced by normal neutron irradiation. Farrell et al. [29] summarized the use of helium implantation in ion irradiation studies and they found that the nucleation rate due to helium implantation increases in the following order: co-implantation, hot pre-implantation, and cold pre-implantation. However, Wolfer and Garner [19] suggested that at moderately large values of the He/dpa ratio, a large number of voids may nucleate, but during subsequent irradiation, the void number density may drop and eventually reach a value independent of the He/dpa ratio. These arguments suggest that cold pre-implantation of helium induces a high initial nucleation rate which may result initially in the suppression of void swelling. However, as the irradiation continues, the number density of voids will reach a saturation value, which is independent of the original number of nucleation sites, and a steady state swelling rate is attained.

There is no significant effect of pre-implanted helium in the ion irradiated microstructure other than bubble formation in the specimens of this study. The result is consistent with other experiments using HT-9 [31]. Although Farrell et al. [29] have reported that pre-implanted helium increases the concentrations of dislocation loops, it is noticed that the maximum effects of helium on dislocation densities should be found at the loop formation stage, i.e., at doses much less than 1 dpa for pure metals and about 1 dpa for swelling-resistant alloys. The lowest dose level of this study is 10 dpa which is too high to easily distinguish effects of helium on dislocation loops.

Helium pre-implantation also showed no significant effect on the precipitate evolution in this alloy. Although some studies [18,29] have shown that helium can retard irradiation-induced phase formation in austenitic stainless steels, there is, unfortunately, no other ferritic steel study that can be used as a comparison. Recently, Maziasz [32] has found a helium-promoted, new phase formation in HT-9 + 2 Ni alloy irradiated in HFIR. This phase is also associated with bubble formation but more work is needed to understand the helium effect on precipitate evolution under irradiation.

5. Conclusions

The results of this study can be summarized as follows:

(a) Helium pre-implantation promotes void formation and growth in the HT-9 specimens following ion irradiation at high temperatures (500 and 600°C).

(b) The overall low swelling (< 0.1%) demonstrates the superior swelling resistance of this alloy. The swelling rate is also very low (< 0.01 %/dpa) which indicates that the void swelling may not be the critical problem for using this alloy in fusion reactor first walls and blankets.

(c) Helium pre-implantation has no significant effects on dislocation loop evolution and precipitate evolution in HT-9 following ion irradiation.

Acknowledgments

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Table 1. Chemical Composition of HT-9 Studied (wt.%)

Fe	Cr	Mo	V	W	Ni	Mn	Si	N	C	P	Cu	S	O
Bal.	12.1	1.04	0.28	0.45	0.51	0.57	0.17	.027	0.20	.016	0.07	.003	.002

Table 2. Cavity Characteristics of HT-9 Bombarded with 14 MeV Ni Ions After Pre-implantation with 100 appm Helium

Irradiation Temp. (°C)	Fluence dpa	Voids $\bar{d}_v$ (nm)	Voids n_v (m^{-3})	$\Delta V/V$ (%)	Helium Bubbles $\bar{d}_b$ (nm)	Helium Bubbles n_b (m^{-3})
400	10	---	---	---	---	---
400	30	---	---	---	---	---
500	10	---	---	---	---	---
500	30	19	$\leq 1 \times 10^{17}$	<< 0.01	1.5	2×10^{22}
500	60	27	1×10^{20}	0.1 (locally)*	---	---
			2×10^{19}	0.02 (average)*	---	---
600	10	---	---	---	2.2	8×10^{21}
600	30	---	---	---	2.3	1×10^{22}

* Voids distributed heterogeneously so that the local swelling is much higher than the average total swelling.

--- None Observed

Figure Captions

Fig. 1. Relationship between the calculated damage profile and helium pre-implanted area in 14 MeV Ni ion irradiated HT-9.

Fig. 2. Helium bubbles in helium doped HT-9 following ion irradiation at 600°C to 10 dpa.

Fig. 3. Voids observed in helium doped HT-9 following ion irradiation at 500°C; 30 dpa (a) and (b), 60 dpa (c) and (d).

Fig. 4. Swelling vs. dose level in helium doped HT-9 following ion irradiation at 500°C.

Fig. 5. TEM microstructures of helium doped HT-9 following ion irradiation to (a) 10 dpa and (b) 30 dpa.

Fig. 6. Critical cavity size determination in helium doped HT-9 following ion irradiation at 500°C ((a) and (b)) and 600°C ((c) and (d)).

Fig. 7. The chi phase particles in the peak damage region of HT-9 following ion irradiation at 500°C to 40, 100 and 200 dpa.

FIGURE 1

14 MeV Ni IONS ON HT-9 TARGET

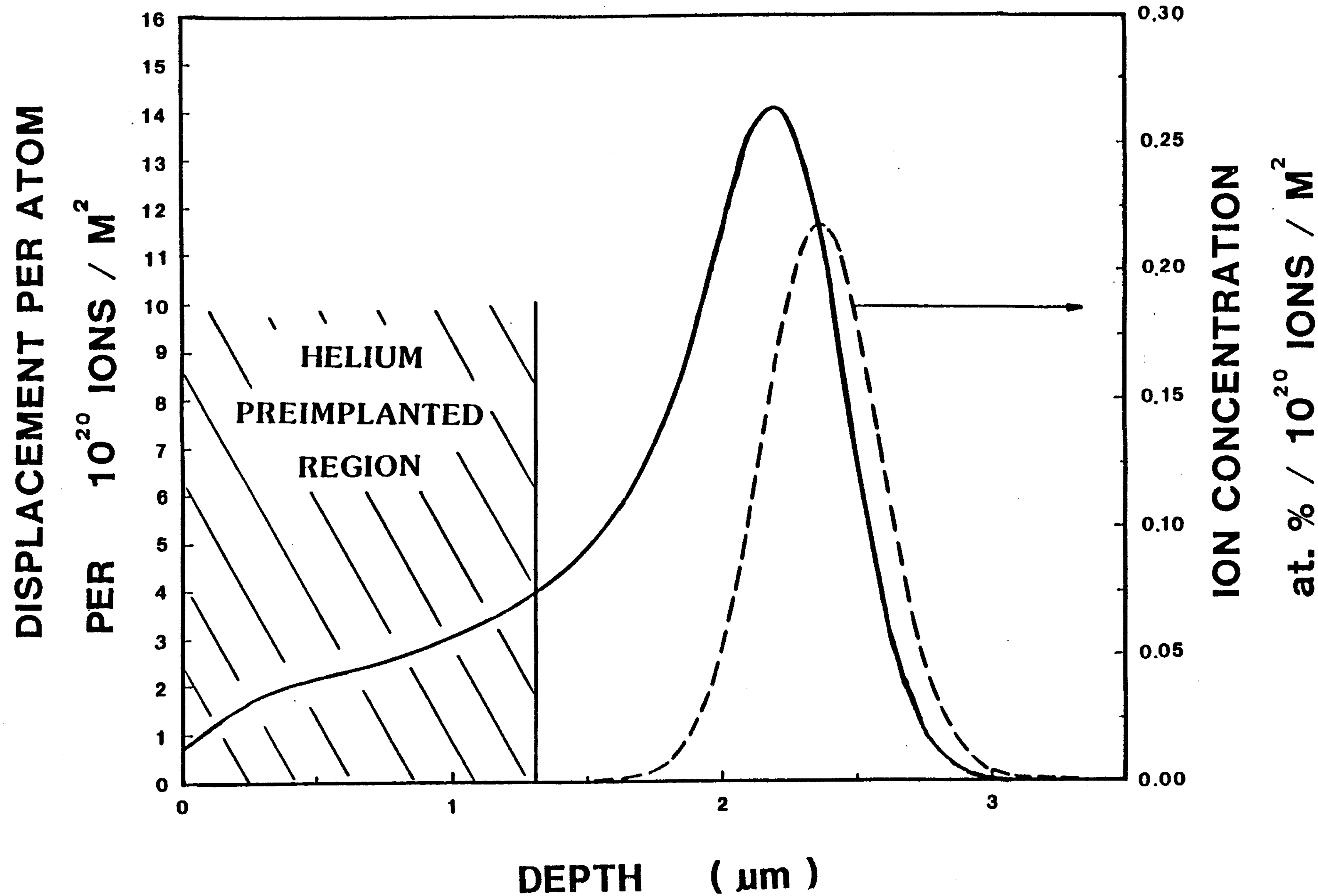
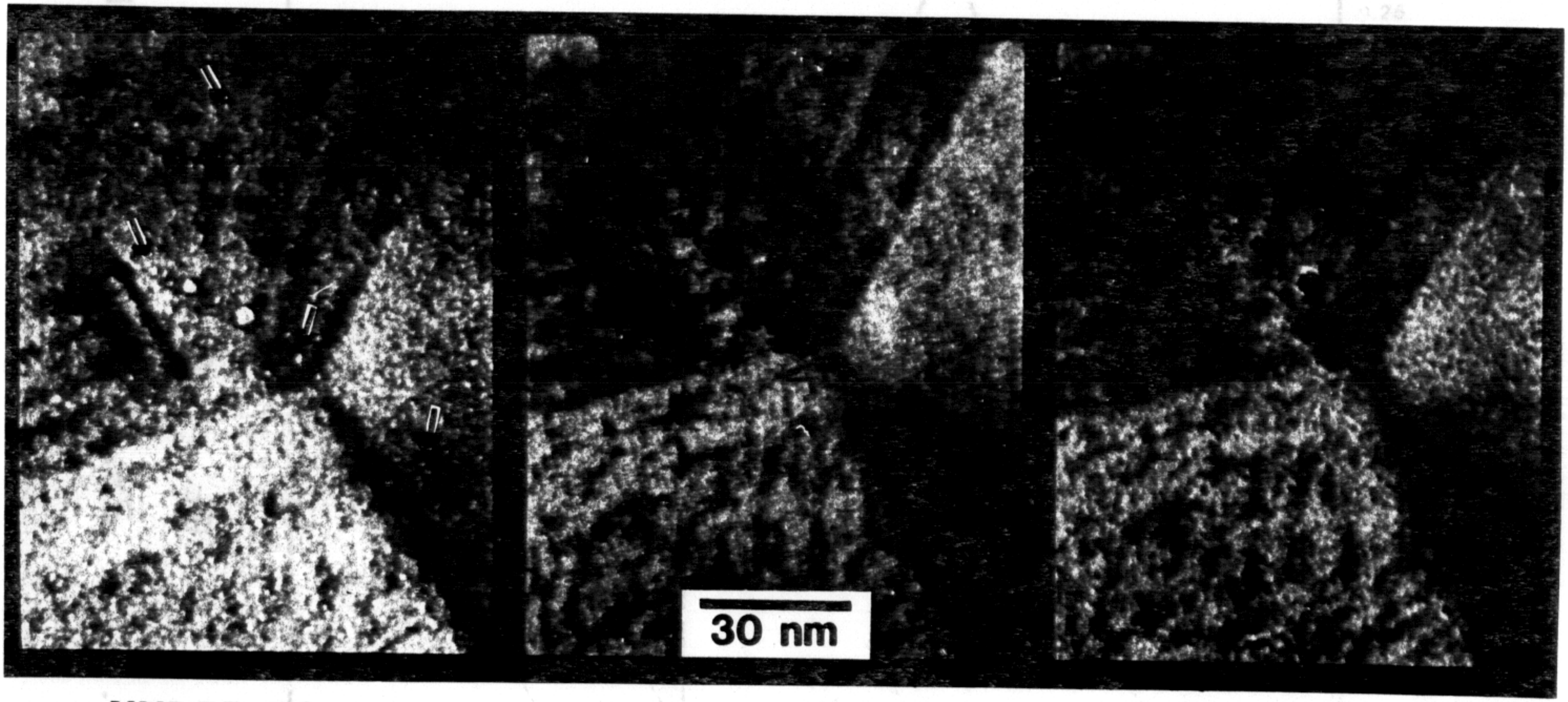


FIGURE 2



UNDER FOCUS EXACT FOCUS OVER FOCUS

FIGURE 3

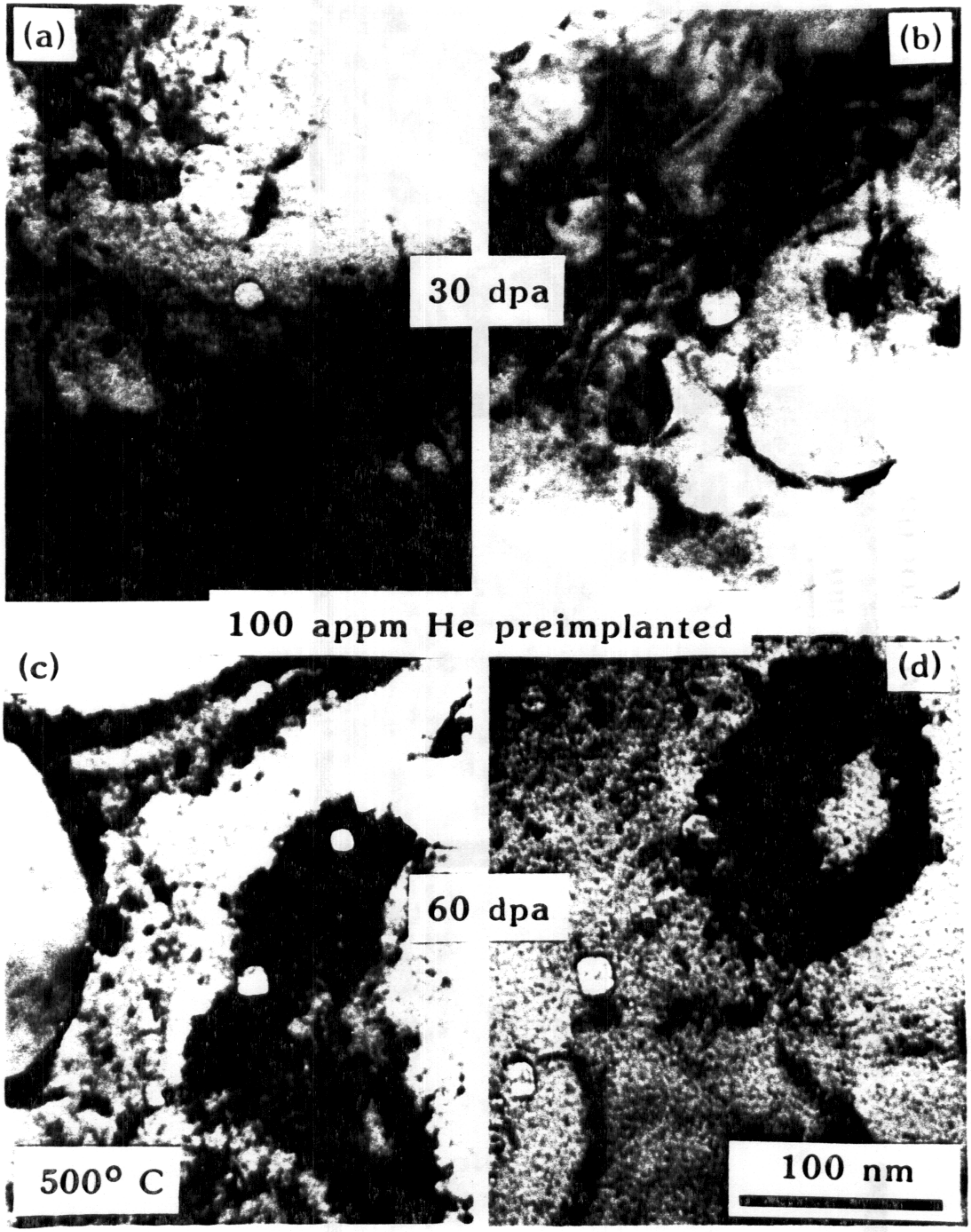


FIGURE 4

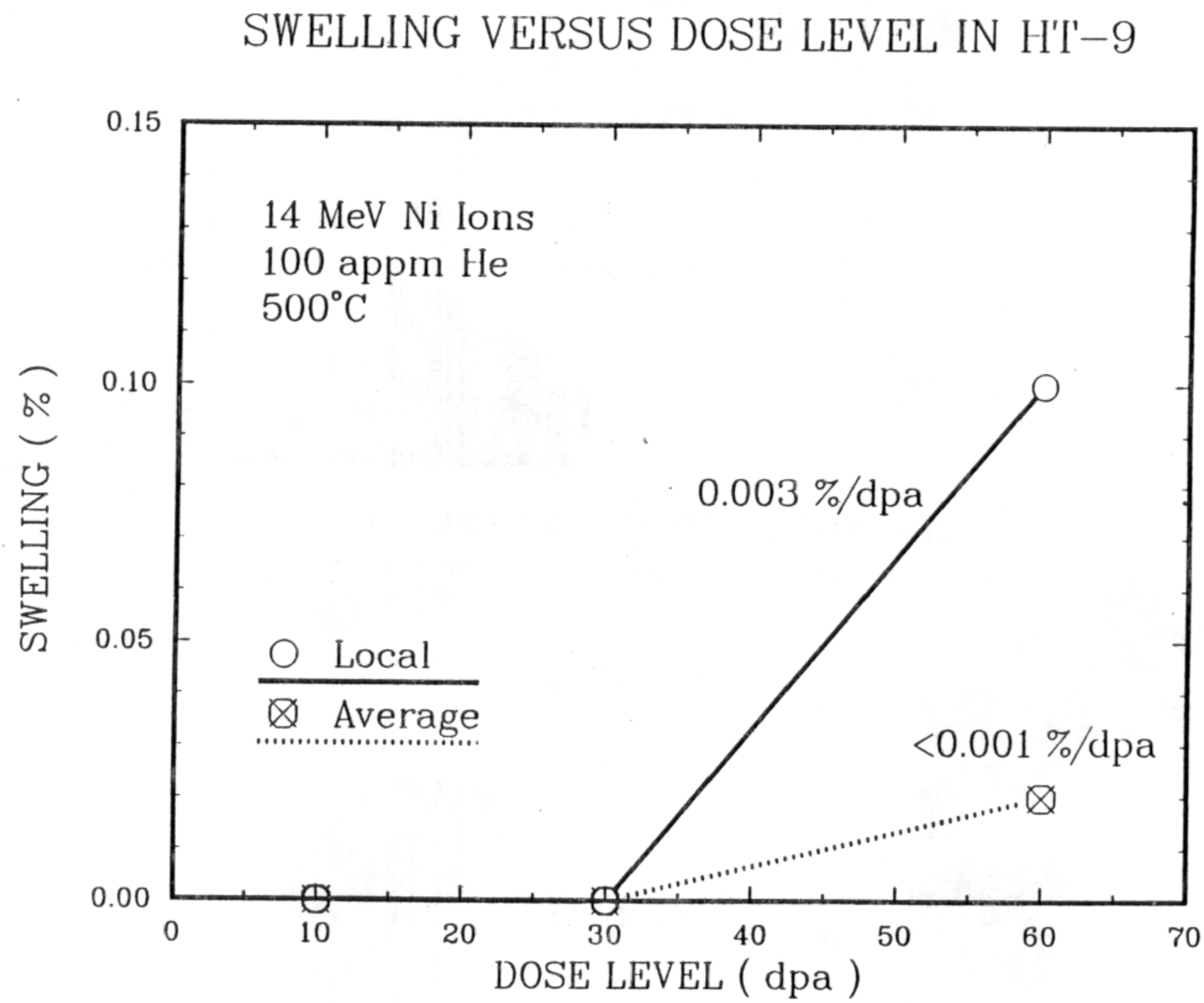
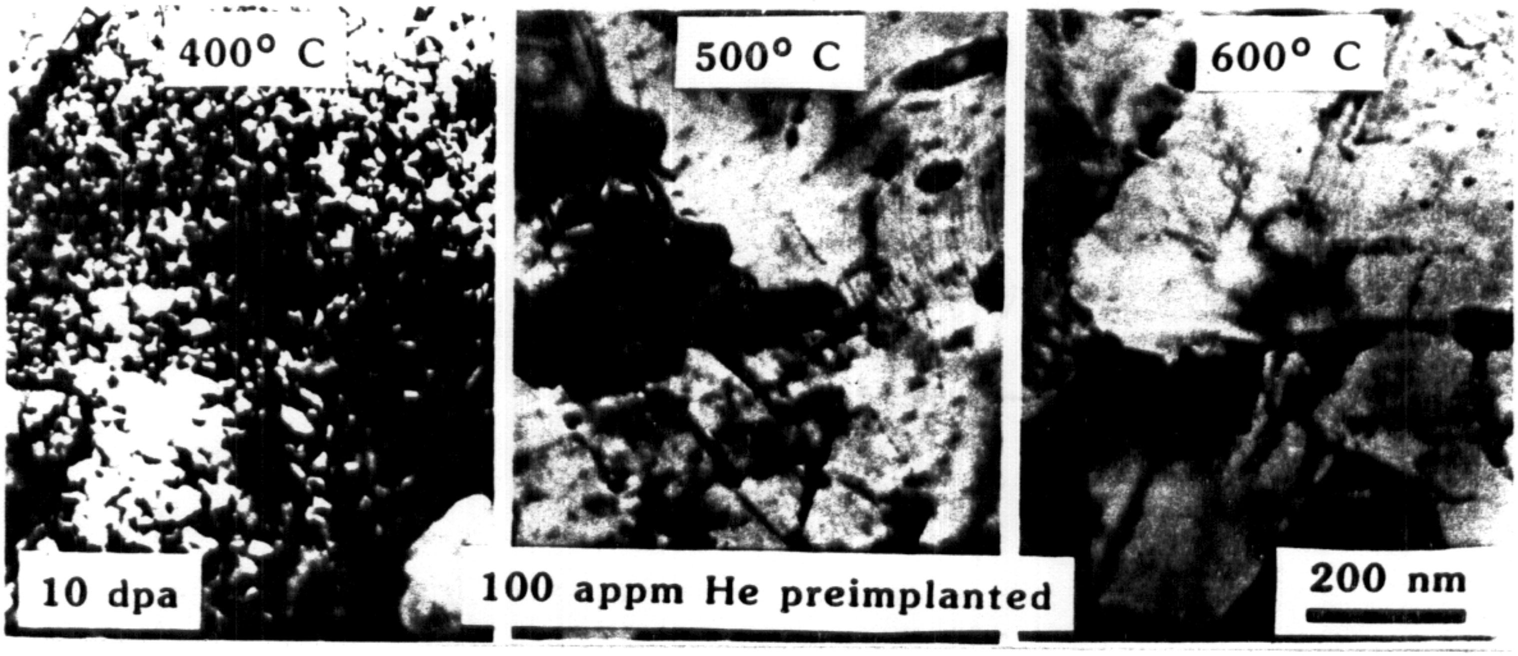
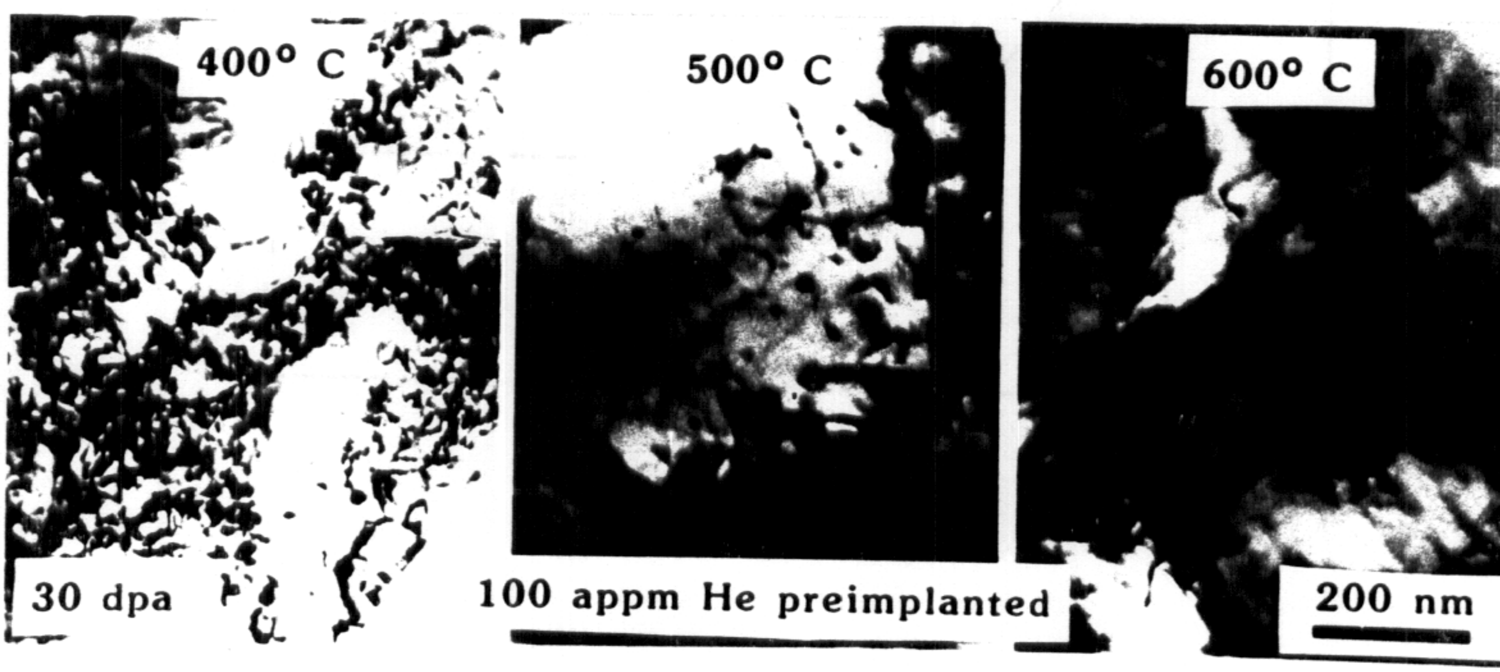

SWELLING VERSUS DOSE LEVEL IN HT-9
14 MeV Ni Ions
100 appm He
500°C
0.003 %/dpa
<0.001 %/dpa
Local
Average
SWELLING (%)
DOSE LEVEL (dpa)
0.15
0.10
0.05
0.00
0
10
20
30
40
50
60
70

FIGURE 5


400° C
500° C
600° C
10 dpa
100 appm He preimplanted
200 nm

a)


400° C
500° C
600° C
30 dpa
100 appm He preimplanted
200 nm

b)

FIGURE 6

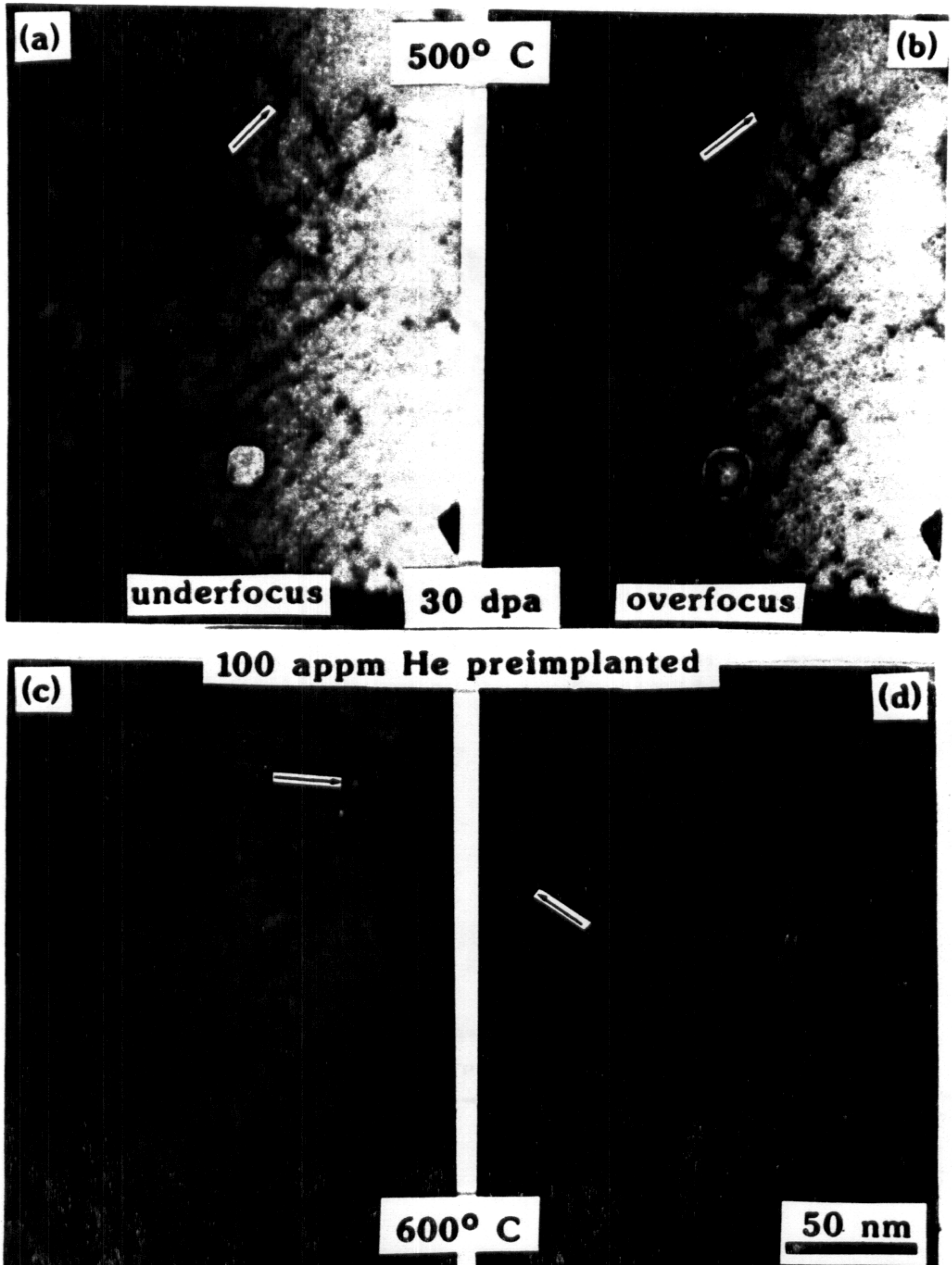

(a)
500° C
(b)
underfocus
30 dpa
overfocus
100 appm He preimplanted
(c)
(d)
600° C
50 nm

FIGURE 7