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DIRECT OBSERVATION OF SOLUTE SEGREGATION TO VOIDS IN A FAST-NEUTRON IRRADIATED Mo/1.0 At% Ti ALLOY

by

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DISCLAIMER

DIRECT OBSERVATION OF SOLUTE SEGREGATION TO VOIDS IN A FAST-NEUTRON IRRADIATED Mo/1.0 At% Ti ALLOY*

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The atom-probe field-ion microscope (FIM) has been employed to study segregation effects, to voids, in a Mo/1.0 at% Ti alloy specimen; this specimen had been irradiated with fast neutrons at ∿700°C, to a fluence of ∿1x10^{22} nvt(energy>1 MeV), in the row 7 midplane position of EBR-II. It had been shown previously, by several other research groups, that this alloy exhibits an enhanced swelling relative to pure Mo when it is fast-neutron irradiated. The results of the present work, on this fast-neutron irradiated alloy, indicated that: (1) titanium does not segregate significantly to voids; (2) the concentration of titanium in solid solution and the spatial distribution of titanium was <u>not</u> affected significantly by the irradiation at 700°C; (3) carbon was <u>not</u> detected in solid solution in either the irradiated or unirradiated specimens; (4) the preceding two results indicate that large-scale resolution of TiC or Mo_2C precipitates had not occurred as a result of the irradiation; and (5) segregation of carbon to a void was detected. The small amount of carbon segregation which had been detected may have been sufficient to enhance the nucleation rate and/or change the sink properties of a void and thus affect the void growth-rate.

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1. Introduction

The body-centered cubic (bcc) alloy Mo/1.0 at% Ti exhibits an enhanced swelling relative to pure Mo when specimens of the alloy and Mo are irradiated with fast neutrons at the same flux and temperature, and to the same fluence.[1-3] Although the composition of the alloy must be related to the enhanced swelling, the mechanism(s) by which the addition of Ti to Mo causes the enhanced swelling is unknown. In the present paper we report on an atom-probe field-ion microscope (FIM) investigation of the nature and extent of the solute redistribution that occurs in a fast-neutron irradiated Mo/1.0 at% Ti alloy. The objective of the study was to elucidate the mechanism(s) responsible for the enhanced swelling of the Mo/1.0 at% Ti alloy relative to pure Mo. Before presenting our experimental results we discuss briefly several aspects of the swelling phenomenon related to solute atoms.

A number of mechanisms for the nucleation and growth of the voids and the interstitial dislocation-loops, that are observed in fast-neutron, ion or electron-irradiated metals, have been proposed. Void-nucleation mechanisms have received considerable theoretical [4,5] and experimental [6,7] attention. It has been suggested, many times, that void nucleation may occur either homogeneously or heterogeneously; in the latter case at defects such as impurity atoms (particularly the interstitial impurities He, C, N or O), depleted zones, precipitates, gas bubbles, etc. Although no single mechanism is able to explain all of the experimental observations it is generally believed that impurities or alloying additions are a major factor in controlling the void nucleation rate.[7]

The void-growth problem has been modeled employing either diffusion-limited kinetics [8] in which the void growth-rate is determined by the vacancy diffusivity or surface-reaction limited kinetics [9] in which the void growth-rate is determined by the ability of the sink-lattice interface to absorb point defects. In both models impurity atoms or alloying additions are expected to have a strong effect on the growth rate. In addition segregation (equilibrium or non-equilibrium) of foreign atoms to the voids or dislocations may have a profound influence on their sink properties by, for example: (1) changing the elastic strain-field around a sink and hence changing the bias of the sink to vacancies or self-interstitial atoms (SIAs) [10]; (2) modifying the surface-reaction kinetics; and (3) inhibiting the climb of dislocations. If tightly-bound SIA-solute atom or vacancy-solute atom complexes form, then a substantial amount of non-equilibrium segregation of the solute to point defect sinks must occur.[11-13]

Observations of massive non-equilibrium solute segregation to voids have been made which were based either on the segregation-induced strain-field around the void or on the irradiation-induced precipitation of second-phase particles.[14-16]* Direct chemical identification of Si segregation to voids in neutron-irradiated aluminum [17] and Si segregation to dislocation loops in ion-irradiated stainless steel [18] had been accomplished employing a scanning-transmission electron-microscope (STEM) equipped with an energy-dispersive X-ray analyzer. Although this method is clearly superior to the more indirect methods previously employed, it is, at present, of limited usefulness in the study of segregation effects in the bcc metals and alloys for the following reasons: (1) the distance between voids in the bcc metals (∿200 Å) is of the order of the minimum diameter which is analyzed chemically; (2) the concentration limit of detectability is 5000 wt. ppm; and (3) the detectable elements range from Na to U in the periodic table, so that one cannot detect the low atomic-number elements such as H, He, C, N and O which are believed to be particularly important impurities in the bcc metals.† Because of the unique

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* See articles by Okamoto and Rehn, and Wiedersich, Okamoto and Lam, in this volume, on radiation-induced segregation.

† The values mentioned in points 1 to 3 are for a JEM-100CX TEMSCAN.

capability of the atom-probe FIM to identify chemically all of the elements, in addition to providing quantitative chemical-analysis on an atomic scale, the atom-probe FIM is ideally suited to study segregation effects in the bcc metals and alloys.

2. Experimental Aspects

2.1. Material and material preparation

The starting material, for the irradiated specimen, consisted of a rod 3cm in length and 0.1 cm in diameter of a Mo/1.0 at% Ti alloy. This rod had been irradiated, along with several other pure Mo and Mo.1.0 at% Ti rods, with fast neutrons at $\sim 700^{\circ}$C to a fluence of $\gtrsim 1 \times 10^{22}$ nvt(energy>1 MeV) in the row 7 midplane position of EBR-II. This rod was thinned to $\gtrsim$0.015 cm in diameter by electropolishing in a solution of 1 part H_2SO_4 to 7 parts CH_3OH by volume at $\gtrsim$10 Vdc. The FIM specimen was then prepared by the drop-off technique [19] employing a 1 normal solution of NaOH in H_2O at $\gtrsim$2 Vac. This specimen was known to contain voids based on an extensive TEM examination of other irradiated rods.

2.2. Experimental procedure

The alloy specimen was examined in our atom-probe FIM; this instrument has been described in some detail in earlier publications [20-24] and hence we only discuss a few important aspects of it in this paper. The atom-probe FIM is basically an FIM which is attached to a specially-constructed time-of-flight (TOF) mass-spectrometer. The mass resolution ($\Delta m/m$) of our instrument is $\gtrsim$1/200 and the lateral chemical spatial-resolution (i.e. the ability to determine the chemistry of two different atoms in or on the surface of an FIM specimen) is a few angstroms while the depth chemical spatial-resolution is determined by the interplanar spacing (<2Å) of the particular atomic plane being examined. The FIM image is observed with the aid of an internal image-intensification system which contains a 3 mm diameter probe hole at its center; see Fig. 1 in Ref.[23]. The FIM specimen is mounted on a liquid-helium-cooled goniometer-stage which provides ±70° rotation about two orthogonal-axes. Thus, the goniometer stage allows any portion of the specimen's surface to be projected into the probe hole for mass analysis, while the FIM image is simultaneously being observed. To chemically analyze a specimen a short (a few nanoseconds in width) high-voltage pulse is applied on top of the best-image voltage; in this manner atoms are pulse field-evaporated in the form of ions; a small portion of the field-evaporation pulse is used to start a digital timer.[25] Those ions that have trajectories that carry them into the probe hole, at the center of the internal image-intensification system, will pass down the TOF tube to a Chevron ion-detector; each ion that arrives at the ion-detector produces a voltage pulse that is used to stop the digital timer. After each field-evaporation pulse a mini-computer reads the TOF and voltage data, computes the mass-to-charge (m/n) ratios and stores the data on magnetic tape. With this computerized system up to 600 TOF events min^{-1} can be automatically analyzed; the computer system operates in real time so that the results are available for interpretation during the course of the experiment.

For the present experiment the basic mode of displaying the data is in the form of an integral profile*; see Fig. 2 in Ref.[23]. An integral profile for a particular alloying-element (e.g. Ti) is obtained by plotting the cumulative number of Ti atoms versus the cumulative number of all the atoms chemically analyzed (e.g. Ti plus Mo); e.g., see Figs. 1, 3, 5-7 in this paper. The first derivative of the integral profile represents the composition profile of the alloying element, since the cumulative number of all

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* We have previously, unfortunately, called this plot a composition profile, but the term integral profile is the more accurate term.

the atoms analyzed is proportional to depth. In the research reported in this paper the average slope of each of the integral profiles presented is approximately a constant; this indicates that the composition profile is approximately flat over the depth analyzed. We will not discuss, in detail, the composition fluctuations in the integral profiles which may be the result of either simple statistical fluctuations or clustering of the Ti atoms.

The data for a typical run of the Mo/1.0 at% Ti were recorded with the atom-probe FIM at a background pressure of $5.2 \cdot 10^{-10}$ torr and at a specimen temperature of 42 K. The partial pressures of the major residual-gas components were: $H_2 = 4.73 \cdot 10^{-10}$ torr; He = $2.0 \cdot 10^{-12}$ torr; $CH_4 = 1.7 \cdot 10^{-11}$ torr; CO = $2.4 \cdot 10^{-11}$ torr; and $CO_2 = 4.0 \cdot 10^{-12}$ torr.

3. Experimental results

3.1. Control experiments on unirradiated specimens

Atom-probe FIM experiments were performed on unirradiated specimens of a Mo/1.0 at% Ti alloy.[23] A typical Ti^{+3} spectrum for the as-received Mo/1.0 at% Ti alloy is shown in Fig. 7 of Wagner et al. [23]; the five isotopes of Ti (Ti^{46}, Ti^{47}, Ti^{48}, Ti^{49} and Ti^{50}) are very clearly resolved from one another; Ti^{+3} isotopes and their relative abundances present a very decisive indicator of Ti which allows us to distinguish it from oxygen; oxygen has only three isotopes (O^{16}, O^{17} and O^{18}) with O^{16} having a natural abundance of 99.8 at% of the three O isotopes. An integral profile of Ti in a Mo/1.0 at% Ti unirradiated specimen is shown in Fig. 1; the depth scale is given in terms of the cumulative number of Mo plus Ti atoms and 10,000 atoms is approximately equal to 200 Å; the latter statement is also true for Figs. 3, 5-7. In the case of this unirradiated specimen (run number 1 in Table 1) the Ti concentration is 0.37 ± 0.06 at%.

The experimentally measured composition for three runs, on three different FIM specimens, is compared with the nominal composition supplied by the manufacturer in Table 1. It is important to note the following: (1) the measured Ti-concentration was consistently less than the nominal concentration supplied by the manufacturer; (2) the measured carbon-concentration was very much smaller than the nominal concentration; and (3) the variation in the experimentally measured Ti-concentration was greater than the expected statistical uncertainty. There are two possible explanations for the low measured Ti-concentration. The first one is that Ti may have also field evaporated as Ti^{+2} and would therefore have been superimposed directly on the Mo^{+4} portion of the mass spectrum. If this is the case than all of the Ti^{+2} would be indistinguishable from the much more abundant Mo^{+4}. We believe that we have ruled out this possibility on basis of the experiments [26] that demonstrated that: (1) the change in the average Mo charge state was small relative to the change in measured Ti-concentration; and (2) there was no obvious correlation between Ti-concentration and the charge state of Mo, i.e. there was no evidence that as the measured Ti-concentration increased that the average Mo charge state and hence the Ti^{+3}/Ti^{+2} ratio increased. These results suggest that it is unlikely that the observed variations in the Ti concentration were due to variations in the Ti^{+3}/Ti^{+2} ratio and that it is unlikely that a significant fraction of the Ti field-evaporated as Ti^{+2}; we note that the discussion of the results presented in Section 3.2 is independent of this conclusion. An alternate and more probable explanation is that the remaining Ti was simply not in solid solution. Considerable metallurgical evidence exists which shows that TiC and Mo_2C precipitates can form in Mo/1.0 at% Ti. [27-29] These precipitates are large and at a low number-density on the scale of an FIM observation and hence the probability of detecting any Ti contained within a TiC precipitate is extremely small. In the course of developing a successful specimen preparation-technique for the Mo/1.0 at% Ti alloy, extensive transmission electron-microscope studies of the electropolished FIM specimens were performed. Occasionally large precipitates

(>1000 Å in diameter) were observed; presumably they were TiC (or Mo_2C) precipitates. This clearly indicated that precipitates were present in the Mo/1 at% Ti used in this experiment. Since the manufacturer's Ti concentration included the Ti contained in the TiC precipitates, there must have been less than 0.9 at% Ti in solid solution. Thus, the Ti concentration measured by the atom-probe FIM technique represents a lower limit on the amount of Ti in solid solution (≈0.25 at%). This argument is also consistent with the experimental observation that less than 0.004 at% C was detected; this shows that the vast majority of the carbon was not in solid solution and that it may reside completely in the TiC (or Mo_2C) precipitates.

The observation (number 3) that the variation in the measured Ti concentration was somewhat greater than the expected statistical variation indicates that the Ti may not be homogeneously distributed in the alloy; it is extremely difficult, from a metallurgical point-of-view, to produce an absolutely homogeneous alloy.† We conclude that the Ti in solid solution was uniformly distributed on a fine scale (<100 Å), but may have been non-uniformly distributed on a scale >100 Å; i.e., the alloy was not homogeneous on a coarse scale.

In order to eliminate the possibility that the carbon detected in a void (see Section 3.2) was due to the decomposition of CO, CO_2 or CH_4, on the surface of the specimen, the following control experiment was performed. An unirradiated Mo/1.0 at% Ti specimen was exposed for periods in excess of 1 hour (which exceeded the time required to chemically analyze the voids) to the typical residual-gases in the FIM in the presence and the absence of the helium imaging-gas. The exposures were made with both the imaging electric-field applied to the specimen or with no imaging electric-field applied to the

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† This statement excludes, of course, the clustering (or negative short-range order) that may occur as a result of the thermodynamics of this alloy.

specimen. Carbon was not detected in any of these control runs. Thus the carbon detected during the analysis of the void (see Section 3.2), must have been present within the specimen and was not an artifact of the vacuum conditions.

3.2. Irradiated specimens

Atom-probe FIM analyses of the region surrounding two voids in the Mo/1.0 at% Ti alloy have been performed. In addition analyses were performed of a region in which no voids were present.

An FIM image showing the first void before and after the atom-probe analysis is presented in Figs. 2(a) and 2(b) respectively; the diameter of this void is between 10 and 20 Å. The area enclosed by the white circle represents the region which was chemically analyzed. Note that the void was still present after the atom-probe analysis and thus the entire analysis was performed on material which was contained within a thin annular-shell surrounding the void. The solid-black circle just below the white circles in Figs. 1(a) and 1(b) is the image of the probe hole. The image contrast shown in Fig. 2 was attributed to a void for the following reasons: (1) the specimen was known to contain voids from transmission electron-microscope studies; (2) the observed contrast effects were different than the contrast effects due to defects other than voids, e.g. vacancies, self-interstitial atoms (SIA), depleted zones, dislocations and grain boundaries; (3) the observed contrast effects were similar to the predicted FIM image-contrast effects from a cavity [30]; and (4) the observed contrast effects were similar to the FIM image-contrast effects reported by Brenner and Seidman [31] for voids in a fast-neutron irradiated molybdenum-specimen.

An integral profile showing the spatial distribution of Ti in the region surrounding this first void is presented in Fig. 3. The measured Ti concentration was 0.4 ± 0.2 at% Ti; this value is within the range of the measured

Ti concentration for the unirradiated alloy (see Table 1). The absence of a statistically-significant difference in the Ti concentration or in the spatial distribution of the Ti in the region around the void versus the unirradiated alloy indicated that a significant amount of Ti segregation to the void had not occurred.

Three successive stages in the examination of the second void are shown in Fig. 4; the diameter of the second void was estimated to be greater than 50 Å. Fig. 4(a) shows the second void prior to the atom-probe FIM analysis and indicates the chemically-analyzed region. The chemical analysis was interrupted and a second observation of the void was made as shown in Fig. 4(b). Note that the void was still present in the specimen which indicated that the material adjacent to this void was still in the process of being chemically analyzed. The atom-probe FIM analysis was then continued until the entire region surrounding the void had been pulse field-evaporated atom-by-atom and hence chemically analyzed. The FIM image of the specimen, taken after the atom-probe FIM analysis had been completed, clearly indicated that the void was no longer present in the specimen as shown in Fig. 4(c).

The Ti integral-profile showing the spatial distribution of Ti in the region surrounding the second void and for the region leading away from this void is presented in Fig. 5. The point at which the atom-probe FIM analysis was interrupted and a visual observation of this void was made is also indicated in this figure. Within the expected statistical-fluctuations the slope of the integral profile in the region of the void had essentially the same as the slope in the region leading away from the void. Thus the Ti was approximately uniformly distributed within the analyzed region. Furthermore the measured Ti concentration was 0.20 ± 0.05 at% Ti; this value is in approximate agreement with the measured range of Ti concentration for the unirradiated alloy (see Table 1). Therefore no evidence of massive segregation of Ti to the voids was found. In addition to titanium, carbon was also detected at a concentration of ∿0.08 ± 0.03 at% C.† The carbon integral-profile is presented in Fig. 6 which shows an approximately uniform distribution of carbon throughout the analyzed region. This indicates that the carbon was probably not contained in a TiC precipitate associated with the void. No evidence of a precipitate was found in the FIM images. The presence of carbon around this void is in sharp contrast with the failure to detect any carbon in solid solution in the unirradiated alloy. This result indicates that either: (1) the carbon had segregated to a pre-existing void during the irradiation; or (2) the carbon was initially incorporated into a void embryo which subsequently grew into a void.

An atom-probe FIM analysis of a region which did not contain any voids was also performed. The integral profile exhibiting the spatial distribution of the Ti remaining in solid solution in the irradiated alloy is shown in Fig. 7. The Ti appears to be approximately uniformly distributed with no evidence that strong clustering of Ti atoms had occurred. A concentration of 0.27 ± 0.05 at% Ti was measured which is within the range of the measured Ti concentration for the <u>un</u>irradiated alloy (see Table 1). This result shows that both the concentration of the Ti remaining in solid solution and the spatial distribution of the Ti was not affected by the irradiation. Thus a very substantial irradiation-induced resolution of Ti from the TiC precipitates had not occurred.* Furthermore no carbon was detected in this run which indicates that carbon was not left in solid solution in the irradiated

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† The failure to detect carbon during the analysis of the first void is not surprising in view of the small total number of atoms detected. Based on a carbon concentration of 0.08 at% the calculated probability of detecting carbon atoms, during the analysis of the first void, was less than 0.5.

* Chang and Perlmutter [29] have shown that TiC precipitates do not thermally dissolve in Mo/1 at% Ti for temperatures less than ∿1600°C.

material. This result confirms the observation that the carbon detected during the analysis of the void must have been associated with the void. The measured composition for the irradiated alloy is summarized in Table 2, while the chemical analysis of the unirradiated alloy is presented in Table 1 for comparison.

The results on the irradiated and the unirradiated Mo/1.0 at% Ti are now summarized:

(1) no evidence of very significant segregation of Ti to voids in the neutron-irradiated alloy has been found;

(2) the concentration of Ti remaining in solid solution and the spatial distribution of the Ti was not significantly affected by fast-neutron irradiation at 700°C;

(3) carbon was not detected in solid solution in either the irradiated or the unirradiated alloy;

(4) results 2 and 3 indicate that large scale re-solution of the TiC or Mo_2C precipitates had not occurred as a result of the irradiation: one cannot rule out some resolution of TiC because of the range of Ti concentrations detected in the unirradiated control specimens; and

(5) segregation of carbon to a void in the irradiated alloy was detected.

4. Discussion

The enhanced swelling of the alloy Mo/1.0 at% Ti relative to pure Mo [1-3] has been attributed primarily to an increase in the void number-density and to a lesser degree to an increase in the average diameter (or volume) of the voids in the alloy.[2] These observations suggest that both the nucleation rate and the void growth-rate is larger in the alloy than in pure Mo.

The results presented in Sections 3.1 and 3.2 imply that the enhanced swelling cannot be attributed to the massive segregation of Ti to voids. The kinetic model-calculation of Johnson and Lam [32] indicated that no enhanced segregation effects can be expected when the binding enthalpies between a solute atom and an SIA and between a solute atom and a vacancy are zero. In addition in the case of voids, the amount of enhanced segregation was also found to be a function of the diameter of the void by Johnson and Lam (see Fig. 5 in [33]); the solute enhancement-factor decreased with decreasing void-diameter. Therefore, our experimental observations may indicate that the values of the binding enthalpies between Ti and the two elementary point defects (the vacancy and the SIA) are vanishingly small. Unfortunately, no measurements of these important binding-enthalpies exist for the Mo/1 at% Ti alloy, although we note that Ti is oversized with respect to Mo [34]; this latter fact may imply that tightly-bound mixed-dumbells[35] do not form in the Mo(Ti) system. It is clear that to make detailed quantitative conclusions, employing the Johnson-Lam model, regarding the absence of massive non-equilibrium-segregation to voids in the Mo(Ti) system, that experimental values of the binding enthalpies are required. Massive nonequilibrium-segregation of Ti to the dislocations can probably be ruled out on the basis of the absence of segregation to voids, although equilibrium segregation of Ti to dislocations remains a possibility. We also note that whatever segregation had occurred it did not seem to effect the ability of the dislocation to climb in the presence of the supersaturation of point defects created by the irradiation conditions. For if segregation had decreased the ability of the dislocations to climb this would have increased the vacancy-SIA recombination rate and hence suppressed void formation (i.e., swelling); since the swelling is enhanced in the Mo/1.0 at% Ti alloy relative to pure Mo we do not believe that there was extensive segregation of Ti to the dislocations.

The small degree of carbon segregation which was detected may have been sufficient to enhance the nucleation rate; and therefore increased the void number-density in the Mo/1.0 at% Ti alloy with respect to pure Mo. In addition the carbon may have had an effect on the sink properties of the void which increased the void growth-rate; this would explain the slightly larger void-diameter in the neutron-irradiated alloy.[2] The carbon that was detected may have come from a small amount of irradiation-induced resolution of MoC (or TiC).

We realize, of course, that the results presented in this paper do not present a definitive answer to the question of the mechanism of swelling enhancement in the Mo/1.0 at% Ti alloy, but they do show that is now possible to obtain quantitative chemical information on an atomic scale that is fine enough such that the present approach will ultimately provide the information required to answer this question.

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Table 1

Comparison of the compostion of a Mo/1.0 at% Ti alloy determined by the FIM atom-probe technique versus the compostion supplied by the manufacturer.

Run Number	Number of Atoms			Concentration (at%)*	
	Mo	Ti	C	Ti	C
1	10,680	40	0	0.37 ± 0.06	< 0.01
2	5,408	28	0	0.52 ± 0.10	< 0.02
3	10,884	30	0	0.27 ± 0.05	< 0.01
Universal Cyclops Steel Corporation's values.				0.90	0.24

* The ± values were calculated from the $\sqrt{N}/\Sigma N$ values where N is the number of atoms detected.

Table 2

The composition of a neutron irradiated Mo/1.0 at% Ti alloy determined by the atom-probe FIM technique.

Region Analyzed	Number of Atoms Detected			Concentration (at%)*	
	Mo	Ti	C	Ti	C
Void Number 1	742	3	0	0.4 ± 0.2	< 0.1
Void Number 2	8,003	16	6	0.20 ± 0.05	0.08 ± 0.03
Void Free Region	10,663	29	0	0.27 ± 0.05	< 0.01

* The ± values were calculated from the $\sqrt{N}/\Sigma N$ values where N is the number of atoms detected.

Figure Captions

Figure 1. The integral profile for Ti in an unirradiated Mo/1.0 at% Ti alloy. The average concentration of the Ti is 0.37 ± 0.06 at% Ti.

Figure 2. Two FIM micrographs showing a void (see white circles) in a neutron-irradiated Mo/1.0 at% Ti specimen; 2(a) is before and 2(b) is after an atom-probe analysis. The solid-black circle below the white circles is the probe hole.

Figure 3. The Ti integral-profile for the region surrounding the first void shown in Fig. 2 for a neutron-irradiated specimen. The average concentration of Ti is 0.4 ± 0.2 at% Ti.

Figure 4. Three FIM micrographs showing a void (see white circles) in a neutron-irradiated specemen; 4(a) is before, 4(b) is during, and 4(c) is after an atom-probe analysis. The solid-black circle below the white circles is the probe hole.

Figure 5. The Ti integral-profile for the region surrounding and leading away from the void shown in Fig. 4 for a neutron-irradiated specimen. The average Ti concentration is 0.20 ± 0.05 at% Ti; on the depth scale 10,000 (Mo + Ti) atoms is approximately equal to 200 Å.

Figure 6. The carbon integral-profile for the region surrounding and leading away from the void shown in Fig. 4 for a neutron-irradiated specimen. The average carbon concentration is 0.08 ± 0.03 at% C; on the depth scale 10,000 (Mo + Ti) atoms is approximately equal to 200 Å.

Figure 7. The Ti integral-profile through a region not containing any voids in a neutron-irradiated specimen. The average Ti concentration is 0.27 ± 0.05 at% Ti; on the depth scale 10,000 (Mo + Ti) atoms is approximately equal to 200 Å.

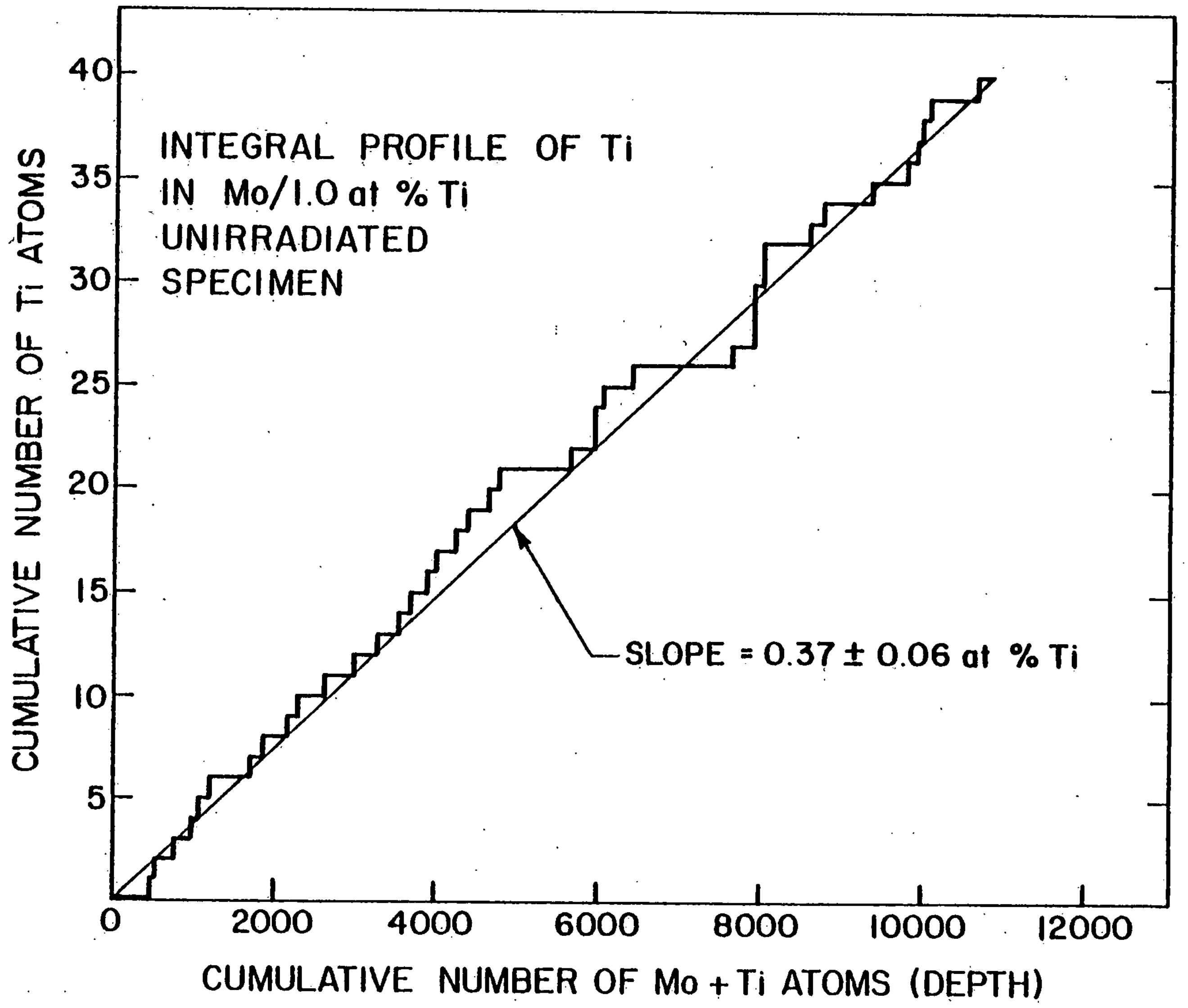
INTEGRAL PROFILE OF Ti
IN Mo/1.0 at % Ti
UNIRRADIATED
SPECIMEN
SLOPE = 0.37 ± 0.06 at % Ti
CUMULATIVE NUMBER OF Ti ATOMS
5
10
15
20
25
30
35
40
0
2000
4000
6000
8000
10000
12000
CUMULATIVE NUMBER OF Mo + Ti ATOMS (DEPTH)

Figure 1

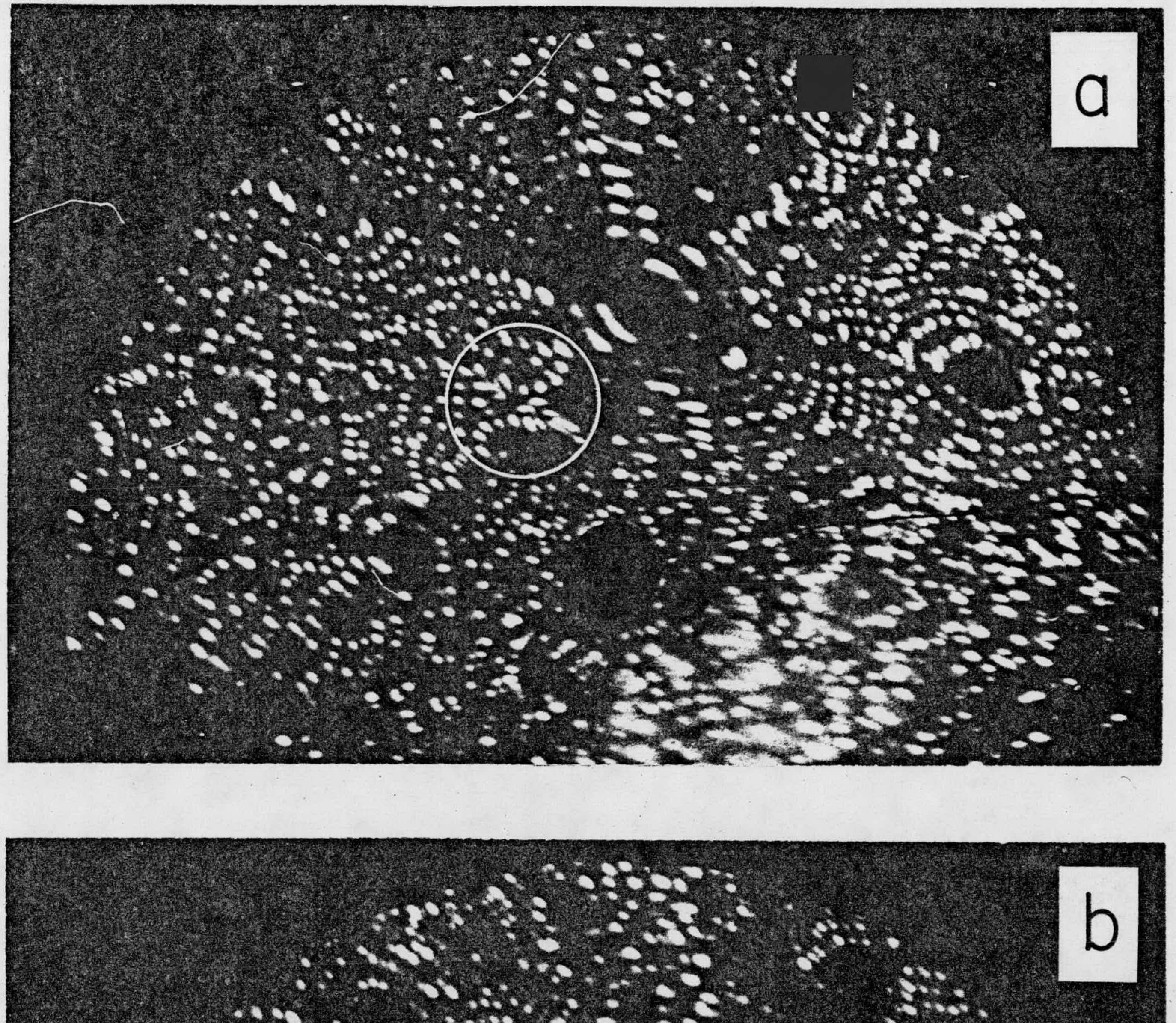

Figure 2

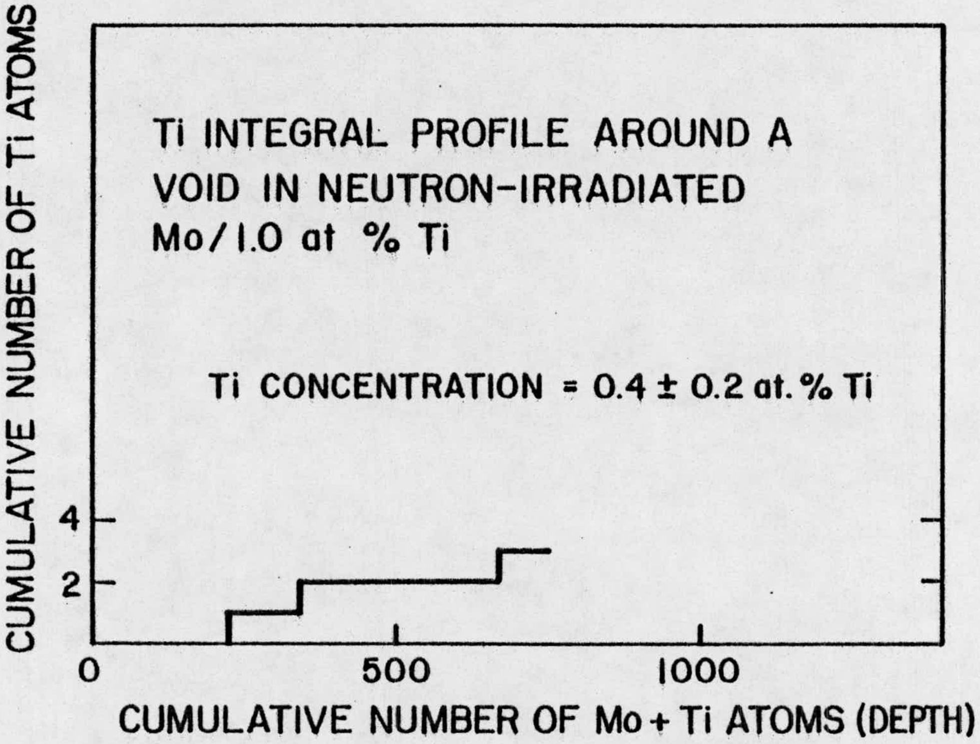
Ti INTEGRAL PROFILE AROUND A VOID IN NEUTRON-IRRADIATED Mo/1.0 at % Ti
Ti CONCENTRATION = 0.4 ± 0.2 at. % Ti
CUMULATIVE NUMBER OF Ti ATOMS
4
2
0
500
1000
CUMULATIVE NUMBER OF Mo + Ti ATOMS (DEPTH)

Figure 3

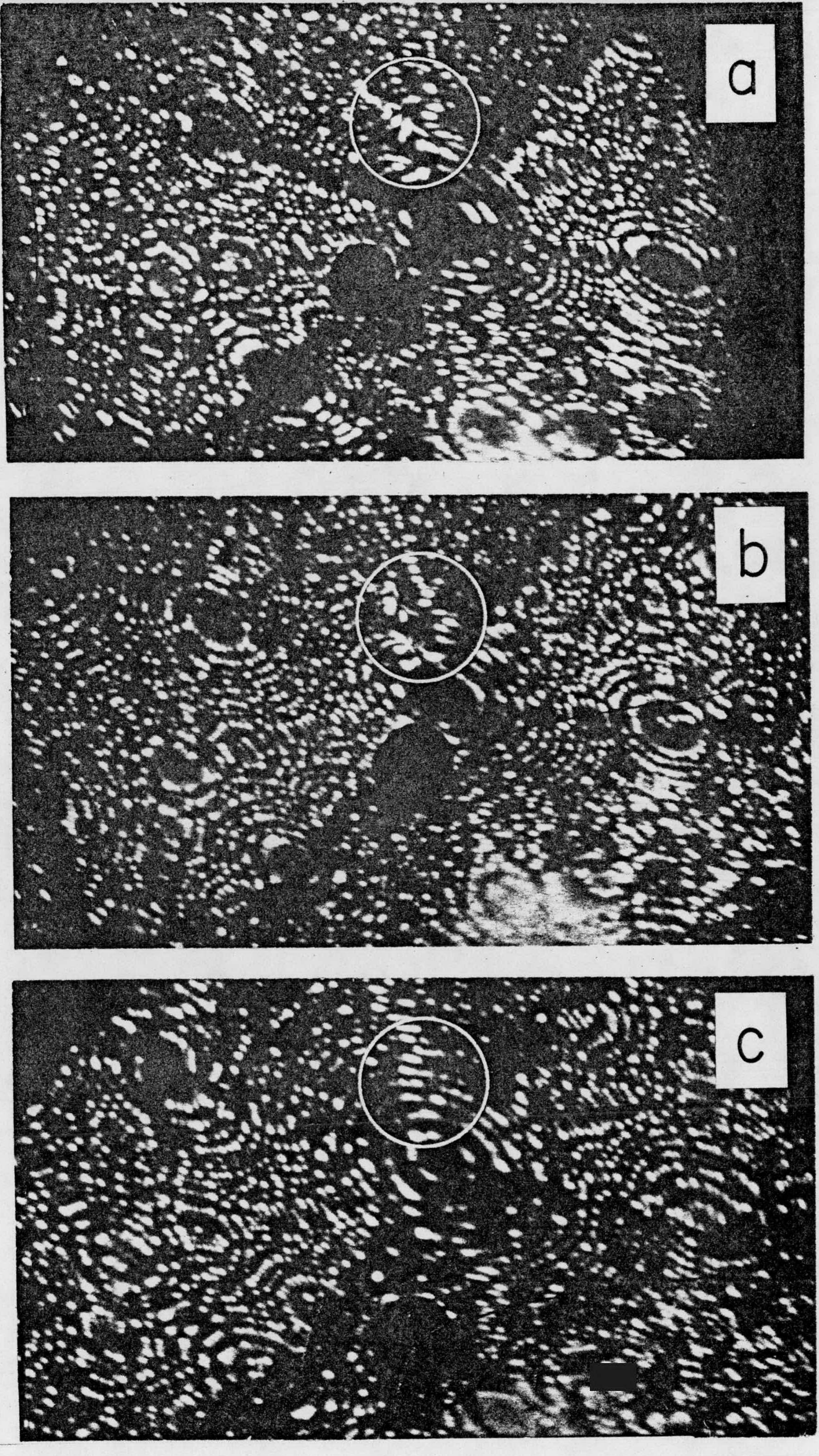

Figure 4

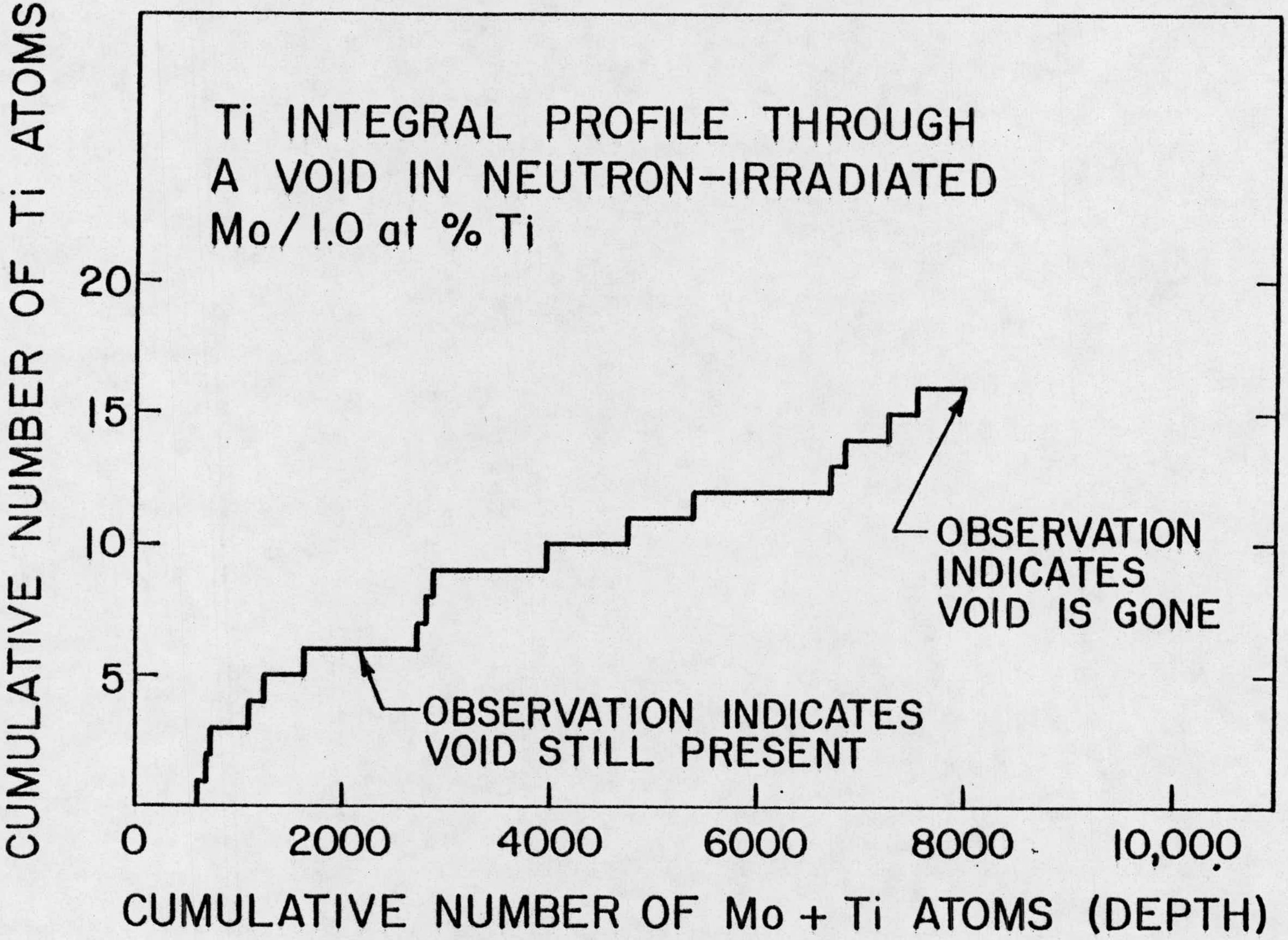
Ti INTEGRAL PROFILE THROUGH
A VOID IN NEUTRON-IRRADIATED
Mo/1.0 at % Ti
CUMULATIVE NUMBER OF Ti ATOMS
20
15
10
5
0
2000
4000
6000
8000
10,000
CUMULATIVE NUMBER OF Mo + Ti ATOMS (DEPTH)
OBSERVATION INDICATES VOID STILL PRESENT
OBSERVATION INDICATES VOID IS GONE

Figure 5

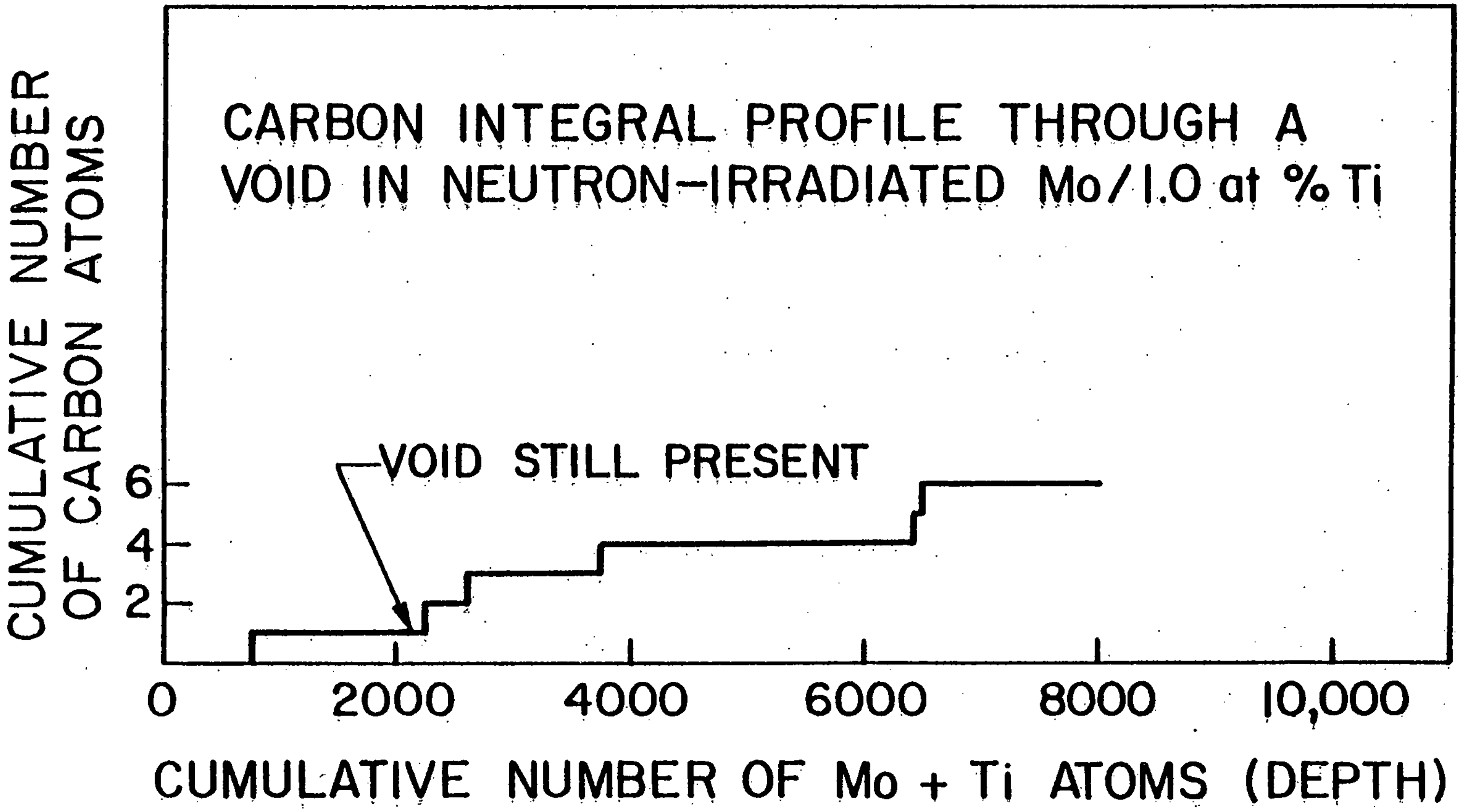
CARBON INTEGRAL PROFILE THROUGH A VOID IN NEUTRON-IRRADIATED Mo/1.0 at % Ti
VOID STILL PRESENT
CUMULATIVE NUMBER OF CARBON ATOMS
2
4
6
0
2000
4000
6000
8000
10,000
CUMULATIVE NUMBER OF Mo + Ti ATOMS (DEPTH)

Figure 6

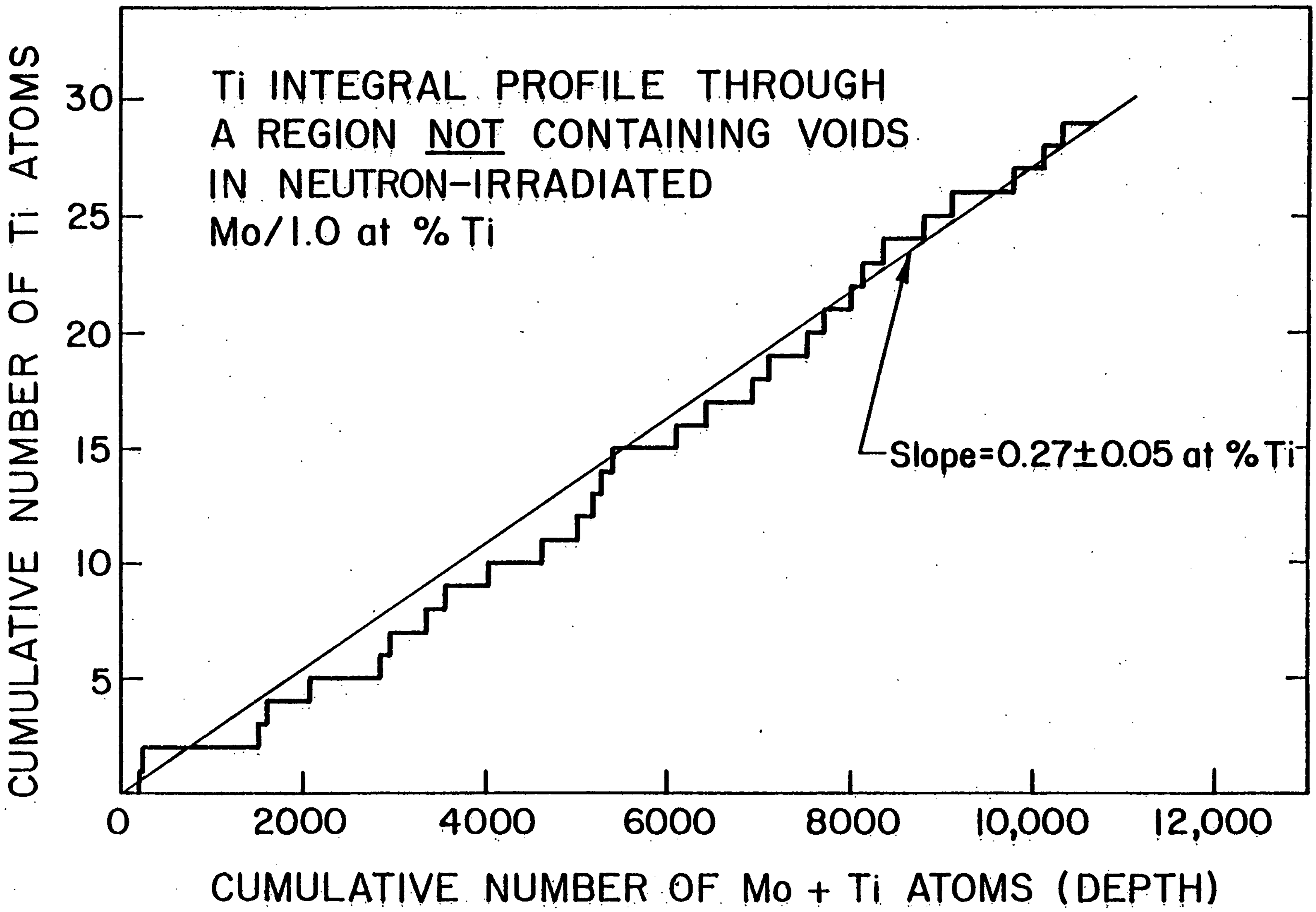
Ti INTEGRAL PROFILE THROUGH
A REGION NOT CONTAINING VOIDS
IN NEUTRON-IRRADIATED
Mo/1.0 at % Ti
Slope=0.27±0.05 at % Ti
CUMULATIVE NUMBER OF Ti ATOMS
30
25
20
15
10
5
0
2000
4000
6000
8000
10,000
12,000
CUMULATIVE NUMBER OF Mo + Ti ATOMS (DEPTH)

Figure 7