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AN ATOM-PROBE FIELD-ION MICROSCOPE FOR THE STUDY
OF THE INTERACTION OF IMPURITY ATOMS OR ALLOYING
ELEMENTS WITH DEFECTS

by

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AN ATOM-PROBE FIELD-ION MICROSCOPE FOR THE
STUDY OF THE INTERACTION OF IMPURITY ATOMS OR
ALLOYING ELEMENTS WITH DEFECTS[†]

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ABSTRACT

A time-of-flight (TOF) atom-probe field-ion microscope (FIM) designed for the study of defects is described. This atom probe features: (1) a variable magnification internal-image-intensification system; (2) a liquid-helium goniometer stage; (3) a low-energy (≤ 3 keV) gas-ion gun for in-situ irradiations; (4) an ultra-high vacuum ($\approx 3 \cdot 10^{-10}$ torr) chamber; (5) a high vacuum ($\approx 10^{-6}$ torr) specimen-exchange device; (6) a Chevron ion detector; and (7) an eight-channel digital timer with a ± 10 nsec resolution for measuring the TOFs. The entire process of applying the evaporation pulse to the specimen, measuring the voltages, and analyzing the TOF data is controlled by a computer. With this system we can record and analyze 600 TOF/min. Results on unirradiated specimens of molybdenum, tungsten, W/25 at% Re, Mo/1.0 at% Ti, Mo/1.0 at% Ti/0.08 at% Zr and a special low swelling stainless steel alloy (LS1A) demonstrate the instrument's ability to quantitatively determine concentrations at the $5 \cdot 10^{-4}$ at fr level and to determine their spatial distribution with a resolution of a few angstroms.

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1. Introduction

The invention [1] of the time-of-flight (TOF) atom-probe field-ion microscope (FIM) has provided us with a unique instrument for the study of the interaction of impurity atoms or alloying elements with point, line or planar defects. The TOF atom-probe FIM consists of a FIM combined with a special TOF mass spectrometer. This spectrometer allows the investigator to chemically identify any atom that appears in a FIM image. Thus, it is now possible both to image the microstructural features of a metal specimen and to measure the mass-to-charge ratios ($\underline{m}/\underline{n}$) of individual atoms from pre-selected regions of a specimen with a spatial resolution of a few angstroms.

The principles and mode of operation of the atom probe can be understood with the aid of fig. 1. A FIM specimen with a radius of ≈ 50 to 500 Å is mounted on a liquid-helium cooled goniometer stage and is maintained at a positive potential of ≈ 5 to 20 kV so that gas atoms (typically helium or neon) are field-ionized over individual atomic sites and are then projected radially outward to produce an image of the atoms on the surface of the metal with the aid of the internal image intensification system (IIIS). In practice the FIM image is observed by means of the front-surfaced glass mirror which is placed at an angle of 45° with respect to the IIIS. The application of a short high-voltage pulse to the FIM specimen results in the field-evaporation of the atoms on the surface of the specimen in the form of positively-charged ions. The ions whose trajectories carry them into the probe hole at the center of the IIIS will subsequently travel down the flight tube to a high gain ($\approx 10^6$) Chevron ion detector. A small fraction of the evaporation pulse is employed to start a high-resolution (± 10 nsec) digital timer [2]; the digital timer is stopped by an output pulse from the Chevron ion detector every time an ion arrives at the detector. The TOFs of the ions and the voltage on the specimen are used to determine the ($\underline{m}/\underline{n}$) ratios of the pulse-field evaporated ions. The operation of the atom probe is controlled on-line by a minicomputer which triggers the high-voltage pulse to the tip, reads in the TOF and voltage data, and then calculates the ($\underline{m}/\underline{n}$) values. The minicomputer operates in real time so that the results can be

displayed immediately on the graphics terminal as the experiment progresses.

Since the first report of the atom probe [1] other instruments have also been reported in the scientific literature and at recent Field Emission Symposia.[†] The major applications of the atom probe, to date, have been to metallurgical problems involving precipitation by Brenner et al. [3,4,5] and by Turner et al. [6,7,8]. The atom probe is ideally suited for the study of the interaction of impurity atoms or alloying elements with lattice defects. The basic reason for this is that by controlled pulse-field evaporation of successive layers [9,10] it is possible to examine the bulk of the specimen and to reconstruct in three dimensions the correspondence between specific microstructural features and the local chemical composition. More specifically the atom-probe FIM is capable of imaging lattice defects such as vacancies [11,12], self-interstitial atoms (SIAs) [15], dislocations [16], grain boundaries [17] and voids [18] and it can also chemically identify both substitutional and interstitial alloying elements or impurity atoms.

In this paper we briefly describe some of the special features of the atom probe we have constructed (§2). We also present the results of a number of experiments which demonstrate the instrument's ability to examine a wide variety of pure metals and metal alloys and to determine the concentration of alloying elements or impurity atoms at the $5 \cdot 10^{-4}$ at fr level (§3).

2. Atom-probe field-ion microscope system

2.1. Time-of-flight mass spectrometer

To determine (m/n) values the voltage on the specimen is measured by an analog-to-digital (A/D) converter. The TOF and voltage data is sent to the minicomputer which then calculates the (m/n) ratios employing the equation

$$m/n = 2e(V_{dc} + \alpha V_{pulse}) (t-t_o)^2/d^2, \quad (1)$$

[†] See Müller and Tsong [13] and Panitz [14] for review articles on the atom probe.

(Panitz et al. [19]) where e is the charge on an electron, V_{dc} is the steady-state imaging voltage, V_{pulse} is the evaporation pulse voltage, α is the pulse factor, d is the flight distance, and $(t-t_0)$ is the actual TOF of the ion. The quantity t is the observed TOF and t_0 is the total delay time (560 nsec). Eq. (1) was derived by equating the kinetic energy of a pulse field-evaporated ion to its potential energy under the assumption that the ion achieves its terminal velocity in a distance that is extremely small compared with d . The controls of the dc, pulse and focusing lens power supplies are coupled together so that V_{pulse} and the lens voltage are always a constant fraction of V_{dc} ; this technique simplifies the calibration procedure and optimizes the resolution of the atom probe [20]. The pulse voltage is typically maintained at $(0.04 \text{ to } 0.20) V_{dc}$ and the lens is maintained at $(0.3 \text{ to } 0.5) V_{dc}$. Below $\approx 0.04 V_{dc}$ the pulser behaves erratically and the continuous dc field-evaporation rate becomes comparable to the pulse-field evaporation rate. For values of $V_{pulse} > 0.20 V_{dc}$ the increased spread in the energy of the field-evaporated ions degrades the mass resolution ($\Delta m/m$) of the spectrometer.

2.2. Internal image-intensification system and focusing lens

The FIM image is observed with the aid of an IIIS (see fig. 1) which consists of a high gain ($\approx 10^3$) Galileo 75 mm diam channel electron multiplier array and a phosphor screen. The entire assembly consisting of the IIIS, focusing lens and front-surfaced glass mirror is attached to two ultra-high vacuum metal bellows arranged so that the distance from the tip of the specimen to the front surface of the IIIS can be varied continuously from ≈ 12 to 100 mm; thus providing an areal magnification change of $> 64\times$.

2.3. Helium-cooled goniometer stage

A liquid-helium cooled goniometer stage has been constructed which has the following features: (1) the specimen can be quickly cooled to cryogenic temperatures to improve the quality and resolution of the FIM image and also to change the diffusivity of point defects; (2) the specimen can be rotated by $\pm 35^\circ$ about two orthogonal axes which intersect at the tip of the specimen so that a selected region of the specimen can be pro-

jected over the probe hole for chemical analysis; and (3) the goniometer stage can be translated in three mutually orthogonal directions to facilitate the alignment of the tip with respect to the probe hole and the low-energy gas ion-gun described in §2.4. Performance tests showed that the FIM specimen is cooled to 30 K within ≈ 20 min and to 13 K within 1 hour with a liquid helium consumption rate of ≈ 1.5 l/hr at 13 K. At temperatures greater than ≈ 30 K the consumption rate drops to ≈ 0.1 l/hr.

2.4. Low-energy gas ion-gun

A low-energy gas ion-gun [21] has been constructed to enable us to irradiate FIM specimens in-situ. In operating the ion gun, a gas such as H_2 , He, Ne, Ar or Xe is continuously bled through a stainless steel cylindrical plasma chamber held at a positive potential. The gas is ionized in the plasma chamber by a magnetically confined 2 mA beam of 30 to 100 eV electrons emitted from a heated tungsten filament. The ions produced in the plasma chamber are extracted, accelerated and focused onto the FIM specimen which is held at ground potential. The ion gun can produce a 4 cm diam beam of 200 to 3000 eV gas ions at a current density of 0.01 to 10 $\mu A/cm^2$ with a gas pressure ranging between $3 \cdot 10^{-6}$ to $5 \cdot 10^{-4}$ torr respectively.

The low energy ions are used to produce a sea of SIAs by the focused collision replacement (RCS) mechanism. As a result of the RCSs the immobile vacancies remain at the irradiated surface and the SIAs are injected into the tip of the FIM specimen. The vacancies in the surface can be removed by the subsequent field-evaporation of several surface layers. The ion gun is also used to implant gas atoms (e.g., hydrogen) in the tip of the FIM specimen.

2.5. The ultra-high vacuum FIM

The primary vacuum pumps are a titanium sublimation pump (TSP) and a 140 l/sec ion pump. The vacuum chamber is initially rough pumped by two sorption pumps used in sequence. A well-trapped 5 cm diam oil diffusion pump is used to pump the main chamber when the atom-probe FIM contains an imaging gas. At all other times the diffusion pump is valved off to prevent possible backstreaming of oil into the vacuum system. Ultimate

pressures of $4 \cdot 10^{-10}$ torr are obtained after baking to 150°C for several hours, while under typical operating conditions the pressure is $1 \cdot 10^{-9}$ torr. The composition of the residual pressure in the vacuum system is measured with a Uthe Technology Inc (UTI) Model 100C residual gas analyzer.

A specimen-exchange air-lock was incorporated into the design of the atom probe to allow the FIM specimen to be replaced without exposing the vacuum system to atmospheric pressure. The pressure in the FIM remains below 10^{-7} torr during the exchange and drops to $3 \cdot 10^{-9}$ torr within 15 min after the exchange port has been closed.

3. Results

In this section we present atom probe results on control specimens of a number of metal alloy systems which we are presently studying. The specific aims of the research program are: (1) the determination of the extent of irradiation induced segregation to voids in fast-neutron irradiated alloys; (2) the measurement of the number density, size distribution, morphology and chemical composition of irradiation induced precipitates; and (3) a determination of the extent of redistribution of alloying elements and/or impurity atoms as a result of fast neutron or heavy ion irradiation. The objective for all three experiments is to try to understand the data obtained in terms of possible implications on the mechanism(s) responsible for the suppression or enhancement of void formation. A strong effort is also being made to determine the role played by gaseous impurities in the nucleation and stabilization of voids.

Before considering the results we discuss how the data is obtained and displayed. As a FIM specimen is field evaporated the voltage is slowly raised by the investigator to maintain a steady pulse-field evaporation rate. After each pulse the computer reads in the TOF and voltage data, computes the $(\underline{m}/\underline{n})$ values and stores the data on magnetic tape. When sufficient data has been accumulated it is displayed on the graphics display terminal in the desired form. At present the two basic modes of display used are: (1) a histogram of the number of events versus the $(\underline{m}/\underline{n})$ ratio; and (2) a composition profile showing the cumulative number of atoms of a given type, within a specified $(\underline{m}/\underline{n})$

range, as a function of the total number of atoms detected.

A schematic illustration of the physical principle behind a composition profile is illustrated in fig. 2. Fig. 2(a) shows a FIM tip formed from a binary alloy of B (solute) in A (solvent). The tip contains a single precipitate which is rich in B; the solute atoms are denoted by the solid black circles. The magnification is adjusted so that during the course of pulse-field evaporation of the specimen all the atoms (A and B) contained within a cylindrical volume element are chemically analyzed by the atom probe. Fig. 3(b) shows the corresponding composition profile; the number of B atoms versus the cumulative number of A plus B atoms. The cumulative number of atoms is proportional to depth (z), since the specimen is chemically analyzed by dissecting atomic planes sequentially; in a typical experiment the specimen is analyzed to a depth of ≈ 250 Å. If the B atoms are distributed uniformly[†] the average slope of the plot in the regions away from the indicated precipitate determines the average composition of the alloy. In the region of the precipitate the average slope of the composition profile yields the precipitate's average composition. The identification of this change in composition as being caused by a precipitate can be confirmed from the microstructural information present in the FIM image. It is also possible to use the composition profiles, in conjunction with the FIM images, to distinguish among: (1) clustering; (2) short-range order (or anti-clustering); (3) composition fluctuations; and (4) pre-precipitation clusters such as Guinier-Preston zones. In this section we use the term cluster to indicate any local composition fluctuation which is obviously greater than the composition fluctuations expected in a random solid solution.

3.1. Molybdenum

The spectrum of a pure molybdenum specimen is shown in fig. 3 for (m/n) ranging from 0 to 100 amu. Molybdenum field evaporates in three charge states (Mo^{+2} , Mo^{+3} and Mo^{+4}); Mo^{+3} is the most abundant state and Mo^{+4} is the least abundant one. Note the

[†] We will use the term uniform to indicate an absence of any gross segregation effects in the volume of space sampled.

presence of the two relatively small peaks of H^+ and He^+ caused by residual hydrogen and helium in the FIM. Fig. 4 shows the Mo^{+2} portion of the spectrum and it is evident that the seven stable isotopes of Mo (^{92}Mo , ^{94}Mo , ^{95}Mo , ^{96}Mo , ^{97}Mo , ^{98}Mo and ^{100}Mo) are resolved; in addition we have obtained isotopic abundances which agreed very well with the handbook values [21,22].

3.2. Tungsten and tungsten/25 at% rhenium

The addition of Re to W strongly suppresses swelling (void formation) in fast neutron irradiated W(Re) alloys [23]. It is our objective to determine how the Re is redistributed as a result of the fast neutron irradiation treatment and to study the irradiation produced precipitates in this alloy. The results on W and W/25 at% Re will serve as a control for some in-progress experiments.

The peaks associated with the five stable isotopes of W (^{180}W , ^{182}W , ^{183}W , ^{184}W , and ^{186}W) are readily distinguished in the W^{+3} spectrum shown in fig. 5(a). The spectrum for the W/25 at% Re alloy is shown in fig. 5(b). The peaks due to the two isotopes of Re (^{185}Re and ^{187}Re) are clearly resolved from the immediately adjacent W isotopes.[†] For both pure W and the W(Re) alloy the isotopic abundances are in good agreement with the handbook values [21,22]. The average composition of the Re in the W(Re) alloy was determined from the composition profiles shown in fig. 6. The average composition for the ^{185}Re isotope is 10.7 at% and for the ^{187}Re isotope it is 14.7 at%; hence the overall Re composition is 25.4 at%. The good agreement between the measured and nominal Re concentration and the FIM image indicates that all the Re atoms are in solid solution. A second conclusion is that there was no evidence for any pre-precipitation. A third conclusion is that the composition profiles exhibit composition fluctuations about the average Re isotope compositions (see fig. 6). To date we have been unable to conclude whether or not there is any evidence for short-range order (anti-clustering) obtainable from the composition profiles.

[†] This demonstrates that $(\Delta m/m)$ is $\approx 1/200$ for our atom probe.

3.3. Molybdenum (titanium) and molybdenum (titanium and zirconium) alloys

Both Mo/1.0 at% Ti and Mo/1.0 at% Ti/0.08 at% Zr (TZM) exhibit an enhanced swelling behavior when compared to molybdenum specimens irradiated under identical conditions to the same fast neutron dose [23,24]. At present we are engaged in a study to determine the role(s) played by the Ti in Mo/1.0 at% Ti and the Ti and Zr in TZM in causing this swelling enhancement. It is interesting to note, by contrast, that Johnston et al. [25] have found Ti and Zr additions to be effective swelling inhibitors in a series of model Fe/Cr/Ni alloys.

The Ti^{+3} spectrum for the as-received Mo/1.0 at% Ti alloy is shown in fig. 6; the five isotopes (^{46}Ti , ^{47}Ti , ^{48}Ti , ^{49}Ti and ^{50}Ti) are very well resolved. The agreement between the measured and the handbook isotopic abundances was found to be reasonable in view of the fact that the sample size only involved 30 Ti^{+3} ions for all five isotopes. The five Ti^{+3} isotopes present a very decisive indicator of Ti which allows us to distinguish it from O; oxygen has only three isotopes (^{16}O , ^{17}O and ^{18}O) with ^{16}O having a natural abundance of 99.8 at%. The concentration of Ti was determined to be 0.28 at%; this represents 28% of the expected 1 at% Ti concentration. There may also be additional Ti atoms which field evaporate as Ti^{+2} and they would, therefore, be superimposed on the Mo^{+4} portion of the spectrum. However, it is emphasized that Chang and Perlmutter [26] have shown that Mo_2C , TiC and ZrC precipitates can form in both Mo(Ti) and TZM. Thus, the remaining 72% of the Ti may alternatively be tied up in the form of TiC precipitates which exist at a number density that is below the limit of detection of the FIM. The Ti observed was found to be uniformly distributed throughout the alloy. This is significant because a comparison of the present results with the results on the irradiated Mo(Ti) specimens should allow us to comment in detail on the changes produced in the spatial distribution of Ti atoms in the alloy as a result of the fast neutron irradiations.

Figure 8 shows a semi-logarithmic plot of the (m/n) histogram of TZM between 29 and 35.5 amu for Zr^{+3} and Mo^{+3} . The isotopes of Zr are ^{90}Zr , ^{91}Zr , ^{92}Zr , ^{94}Zr and ^{96}Zr ; hence there is an almost exact overlap with ^{92}Mo , ^{94}Mo and ^{96}Mo . The ^{90}Zr and ^{91}Zr isotopes have isotopic abundances of 51.46 and 11.23 at% respectively; hence they constitute 62.69 at% of the five Zr isotopes. We also observed $^{90}Zr^{+2}$ at 45 amu; hence the total Zr concentration is 0.13 at% (based on the isotopic abundance of ^{90}Zr) which is in reasonable agreement with the nominal Zr concentration.

The composition profiles of Ti and Zr in TZM are shown in fig. 9; each step in the ordinate scale corresponds to single Ti or Zr atom. The Ti atoms observed were distributed uniformly throughout the specimen; from the slope of the composition profile the Ti concentration is 0.26 at%. This value represents 26% of the nominal Ti concentration in TZM. The comment made with respect to the difference between the measured and the nominal Ti concentration in the Mo(Ti) alloy also applies to TZM. All the Zr atoms detected were contained in a single cluster; this Zr cluster was detected just before the FIM tip failed and it may have contributed to the failure of the tip. These control experiments on TZM demonstrated that the Ti and Zr alloying elements in the <0.5 at% range could be easily detected and that the techniques developed to date should work reasonably well for the neutron-irradiated specimens.

3.4. Low swelling stainless steel alloy (LS1A)

A low swelling stainless steel alloy (LS1A) developed at the Oak Ridge National Laboratory (ORNL) by Bloom et al. [27] has been analyzed. To date we have restricted our work to unirradiated specimens of this alloy because of the high radioactivity of the neutron irradiated specimens. The alloy LS1A contains 2.06 at% Si and 0.16 at% Ti as swelling inhibitors. It is the purpose of the present study to determine the role played by these swelling inhibitors.

All the alloying elements (Fe, Cr, Ni, Mn, Mo, C, Si and Ti) in LS1A were readily identified and the concentrations measured by the atom probe technique are in good agreement with the chemical composition as supplied to us by ORNL. A portion of the LS1A

spectrum is exhibited in fig. 10; the isotopes $^{50}\text{Cr}^{+2}$, $^{52}\text{Cr}^{+2}$, $^{54}\text{Fe}^{+2}$, $^{55}\text{Mn}^{+2}$, $^{56}\text{Fe}^{+2}$, $^{58}\text{Ni}^{+2}$ and $^{60}\text{Ni}^{+2}$ can be clearly identified in the 24 to 34 amu portion of the spectrum. In addition, an analysis of the spatial distribution of each alloying element revealed the existence of a cluster consisting of three nickel atoms, three silicon atoms, two carbon atoms, two titanium atoms, two molybdenum atoms, one manganese atom and one iron atom. It is highly improbable for such a cluster to be a simple statistical fluctuation.

4. Conclusions

In this paper we have described a computer controlled atom-probe FIM which is suited for the study of the interaction of impurity atoms or alloying elements with defects. Detailed results were presented on unirradiated specimens of Mo, W, W/25 at% Re, Mo/1.0 at% Ti, Mo/1.0 at% Ti/0.08 at% Zr and a low swelling stainless steel alloy (LS1A) which demonstrate that both the concentration and the spatial distribution (on an angstrom scale) of each alloying element can be determined quantitatively at levels as low as $5 \cdot 10^{-4}$ at fr. We are presently applying this technique to fast-neutron irradiated specimens of the above alloys in an attempt to solve the problems enumerated at the beginning of §3.

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Figure Captions

Figure 1: A schematic diagram of the TOF atom-probe FIM. Shown at the top of the diagram are the internal elements of the atom probe including the FIM specimen, the internal image intensification system, the focusing lens, the viewing mirror and the Chevron ion detector. The specimen voltage system and the digital timer, shown in the lower portion of the figure, are operated automatically by a Nova 1220 minicomputer which is interfaced to a Tektronix 4010 graphics display terminal and a 4610 hard copy unit so that the data can be plotted on-line during the course of an experiment. With this automated system we can presently record and analyze 600 TOF/min.

Figure 2: During the course of an atom-by-atom dissection of a FIM specimen the atom probe determines the distribution of chemical species in an approximately cylindrical volume element of the specimen as shown in 2(a). A plot of the number of B atoms in the binary alloy consisting of A (solvent) and B (solute) atoms versus the total number of atoms (A plus B) detected yields a composition profile. The presence of a local composition variation produced by a precipitate rich in B results in a change of slope of the composition profile; this is illustrated in 2(b).

Figure 3: Spectrum of pure molybdenum at a pressure of $5 \cdot 10^{-9}$ torr, a specimen temperature of ≈ 60 K and with the probe hole in the IIIS near the (110) pole. The calibration parameters used were $\alpha = 1.482$, $t_0 = 0.56$ μ sec and $d = 2.213$ m.

Figure 4: The Mo^{+2} portion of the Mo spectrum exhibited in fig. 3. The total number of Mo^{+2} events in this histogram is 696.

Figure 5a: The W^{+3} spectrum of Westinghouse as-received tungsten pulse-field evaporated at ≈ 25 K for V_{dc} varied continuously from 13 to 15 kV. The ions were collected from the (551) plane and at a pressure of $6 \cdot 10^{-10}$ torr. The calibration parameters used were $\alpha = 2.0$, $t_0 = 0.56$ μ sec and $d = 1.6003$ m. The total number of W^{+3} events in this histogram is 6045.

Figure 5b: The W^{+3} and Re^{+3} spectrum of W/25 at% Re thermocouple wire. The spectrum was recorded at ≈ 25 K at a pressure of $5 \cdot 10^{-9}$ torr. The calibration parameters used were

$\alpha = 1.482$, $t_o = 0.56$ μsec and $d = 2.232$ m. The total number of W^{+3} and Re^{+3} events in this histogram is 1755.

Figure 6a: A composition profile for the ^{187}Re isotope in the W/25 at% Re alloy. The number of events in the range 62.3 to 62.6 amu are plotted as a function of the cumulative number of events in the W/Re^{+3} spectrum from 59.9 to 65 amu.

Figure 6b: A composition profile for the ^{185}Re isotope in the W/25 at% Re alloy. The number of events in the range 61.6 to 62.0 amu is plotted as a function of the cumulative number of events in the W/Re^{+3} spectrum from 59.9 to 65 amu.

Figure 7: This spectrum was recorded with the atom probe at a pressure of $5.2 \cdot 10^{-10}$ torr and with the specimen at a temperature of 42 K. The voltage range was $V_{dc} = 5,600$ to 9,400 V and the calibration parameters were $\alpha = 1.2$, $t_o = 0.56$ μsec and $d = 2.218$ m.

Figure 8: This spectrum was recorded with the atom probe at a pressure of $4.5 \cdot 10^{-10}$ torr and a specimen temperature of 35 K. The voltage range was $V_{dc} = 12,200$ to 13,300 V and the calibration parameters were $\alpha = 1.2$, $t_o = 0.56$ μsec and $d = 2.218$ m. Note that the ordinate scale is logarithmic.

Figure 9: Composition profiles for Ti and Zr in TZM.

Figure 10: The 24 to 34 amu portion of the LS1A spectrum. The main alloying elements (Fe, Ni and Cr) as well as one minor alloying addition (Mn) are seen in this part of the spectrum. This spectrum was recorded at a pressure of $1 \cdot 10^{-9}$ torr and a specimen temperature of 50 K. The voltage range was from $V_{dc} = 16,600$ to 19,900 V and the calibration parameters were $\alpha = 1.482$, $t_o = 0.56$ μsec and $d = 2.227$ m.

FIG. 1

FIG. 2

FIG. 3

FIG. 4

FIG. 5

FIG. 6

FIG. 7

FIG. 8

FIG. 9

FIG. 10