Microstructural Characterization of Selected AEA/UCSB Model FeCuMn Alloys

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Manuscript Completed: June 1995
Date Published: June 1996

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Prepared for
Division of Engineering Technology
Office of Nuclear Regulatory Research
U.S. Nuclear Regulatory Commission
Washington, DC 20555-0001
NRC Job Code L1098

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Abstract

A set of 22 model ferritic alloys was purchased as part of a collaborative research program by the AEA Harwell Laboratory and the University of California at Santa Barbara. Nine of these alloys were selected by the Oak Ridge National Laboratory for use in a series of ion irradiation experiments investigating dispersed barrier hardening. These nine alloys contain varying amounts of copper, manganese, titanium, carbon, and nitrogen. The alloys have been characterized by transmission electron microscopy in the as-received condition to provide a baseline for comparison with the irradiated specimens. A description of the microstructural observations is provided for future reference. This summary focuses on the type and size distributions of the precipitates present; grain size and dislocation measurements are also included.

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Acknowledgments

The authors would like to acknowledge their collaborators: Profs. G. R. Odette and G. E. Lucas from the University of California, Santa Barbara, and Dr. W. J. Phythian from the AEA Harwell Laboratory in the United Kingdom for supplying the material used in this work; and Dr. J. Cawley from Sheffield City Polytechnic in the United Kingdom for preparing the alloys.

The continuing financial support of the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission, is gratefully acknowledged. Contributions to this work were supported in part by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Lockheed Martin Energy Systems and by an appointment of P. M. Rice to the Oak Ridge National Laboratory Postdoctoral Research Associates Program administered jointly by the Oak Ridge National Laboratory and the Oak Ridge Institute for Science and Education.

Foreword

The work reported here was performed at the Oak Ridge National Laboratory (ORNL) under the Heavy-Section Steel Irradiation (HSSI) Program, W. R. Corwin, Program Manager. The program is sponsored by the Office of Nuclear Regulatory Research of the U.S. Nuclear Regulatory Commission (NRC). The technical monitor for the NRC is M. G. Vassilaros.

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Microstructural Characterization of Selected AEA/UCSB Model FeCuMn Alloys*

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Introduction

As part of an ongoing collaboration, researchers at the AEA Harwell Laboratory in the United Kingdom and the University of California at Santa Barbara (UCSB) acquired a set of 22 model ferritic alloys for use in irradiation experiments investigating embrittlement of reactor pressure vessel (RPV) steels. A subset of nine of these alloys was selected by the Heavy-Section Steel Irradiation (HSSI) Program at the Oak Ridge National Laboratory (ORNL) for use in a series of ion-irradiation experiments. The alloys were supplied to ORNL by UCSB.

The alloys were prepared in the United Kingdom at the Sheffield City Polytechnic, and some characterization of the alloys was done by AEA Harwell.^{1,2} In order to provide a complete baseline for comparison with the irradiated samples, a detailed examination of the nine alloys selected by the HSSI Program was carried out by transmission electron microscopy (TEM). This work has focused on the type and size distributions of the precipitates and inclusions observed in the alloys; a limited number of measurements of dislocation density were also carried out. Estimates of grain sizes were obtained by TEM; these were generally consistent with the values reported by AEA Harwell.

The use of model alloys provides a valuable method of isolating the effects of specific compositional changes on the irradiation response of materials. The relative simplicity of their microstructures can highlight radiation-induced changes and elucidate the microstructural basis of hardening and embrittlement. The nine alloys discussed in this report were selected primarily to investigate the role of copper in materials that had both low and high levels of interstitial solutes (C and N). Ion irradiations are being conducted to doses of 0.01, 0.1, and 1.0 displacements per atom, and TEM and hardness measurements are being used to determine the relative hardening efficiency of the radiation-induced defects.

Alloys Selected for Examination

The full set of 22 AEA/UCSB model alloys encompassed systematic variations in copper, manganese, titanium, carbon, and nitrogen content. A high-purity iron from Blyth Metals Ltd. provided the base material. All alloying, including the nitrogen, was made by addition. Each ingot was heat treated at 1100°C after casting and then hot forged to yield a billet of approximately 20 x 80 x 400 mm.

The compositions of the nine alloys selected by ORNL are given in Table 1, along with their alloy designations and the base heat treatment carried out at UCSB. The solution treatment at 775°C was intended to put all the copper in solution in the α -phase field, and the quench was carried out to maintain a high level of dissolved copper. A short temper at 450°C was intended to precipitate carbon in the form of dispersed Fe_3C or M_3C intergranular carbides.

*Research sponsored by the Office of Nuclear Regulatory Research, Division of Engineering, U.S. Nuclear Regulatory Commission, under Interagency Agreement DOE 1886-8109-8L with the U.S. Department of Energy under contract DE-AC05-84OR21400 with Lockheed Martin Energy Systems.

The UCSB heat treatment was somewhat different than the base heat treatment applied to these same alloys by AEA Harwell. The Harwell solution treatment was similar, 16 h at 770°C, but their material was water quenched.^{1,2} It is likely that the Harwell heat treatment maintained somewhat higher levels of copper (and possibly nitrogen) in solution. One potential difference between the two alternate heat treatments was exposed by a short-term aging study of alloy VM397. This will be discussed below.

Table 1. Alloy designations and compositions

Alloy Number	N (appm)	Cu	Mu	Composition (wt %)	
				C	Ti
VM348	5	---	---	---	---
VM349	80	---	---	---	---
VM350	120	---	---	---	---
VM390	20	0.51	0.06	<0.005	0.002
VM397	20	0.91	<0.01	<0.01	<0.01
VM399	120	0.51	0.01	<0.01	<0.01
VM387	10	0.51	0.05	0.17	0.003
VM360	10	0.89	1.03	<0.003	---
VM373	100	<0.01	0.01	<0.003	0.3

As-Received Heat Treatment:
 Solution treated at 775°C for 17 h.
 Quenched in salt bath to 450°C and held for 3 min.
 Air cooled.

Sample Preparation and TEM Examination

Electropolishing Conditions

The specimens were punched as 3-mm discs from 0.5-mm-thick coupons and were then ground and sanded to 0.1 mm (4 mils) to minimize the amount of ferromagnetic material later placed in the TEM. The specimens

*G. R. Odette, G. E. Lucas, R. D. Klingensmith, R. E. Stoller, and W. J. Phythian, "On the Effect of Flux and Composition on Irradiation Hardening at 60°C," presented at the 17th ASTM Symposium on the Effects of Radiation on Materials, June 1994; to be published by ASTM.

were then electrochemically thinned to electron transparency using a chilled perchloric electrolyte in a twin jet polisher. The composition of the electrolyte was: 600 mL methanol, 340 mL butyl cellosolve, 60 mL perchloric acid, and 5 mL glycerine. The electrolyte temperature was maintained at -55°C throughout the polishing, and the electrolyte was stored in a freezer between uses to limit chemical reactions between the electrolyte and the material removed from the specimens.

TEM Examination

The TEM examination was carried out using primarily a Philips CM30 microscope operated at 300 keV. The microscope had an energy dispersive X-ray (EDX) spectrometer capable of detecting elements with $Z > 11$. All the TEM micrographs appearing in this report were acquired with this microscope. Most of the information about elemental contents of the small inclusions, listed in the third column of Tables 2 through 10, was acquired with this microscope. Some light element detection was carried out using a thin-window EDX detector attached to a Philips CM12 microscope operated at 120 keV. The specimen thicknesses used to calculate precipitate and dislocation densities were determined using the X-ray fluorescence method, as described by Kesternich.³

Aging Study of Alloy VM397

In order to provide a point of comparison between the UCSB and AEA base heat treatments, alloy VM397 (Fe-0.9%Cu) was aged at 550°C for times up to 15 h. The formation of copper precipitates during aging hardens this material.^{4,5} A similar aging experiment had been carried out by Harwell; they found a hardening peak in VM397 at about 16 h, followed by softening as the precipitates overaged.¹ As shown in Figure 1, the time to reach peak hardening was reduced to about 10 h in the material that was heat treated at UCSB. This is consistent with some copper precipitation beginning during the 450°C temper and slow air-cooling, while the Harwell water quench maintained a greater fraction of the copper in solution.

Figure 1. Change in Vickers hardness (200-g load) as a function of annealing time at 550°C for alloy VM397.

Description of As-received Alloy Microstructure

The major microstructural observations for the nine alloys are summarized in Tables 2 through 10; they are generally consistent with expectations of clean model alloys. Although TEM measurements of grain size are less accurate than conventional metallography, our observations are similar to those reported by Harwell.¹ The grain sizes were generally large, $\sim 100 \mu\text{m}$. Exceptions were VM348, which exhibited a duplex structure; VM390, which exhibited a finer grain size due to many low-angle boundaries; and VM360 and VM373, which both showed slightly smaller grain sizes of $\sim 50 \mu\text{m}$. Detailed dislocation measurements were not made on all alloys, but the dislocation densities that were measured in VM348, VM349, and VM350 were quite low, $\sim 10^{13} \text{ m}^{-2}$.

The total precipitate density was low in most of the alloys, typically 1 to $7 \times 10^{18} \text{ m}^{-3}$. The precipitates were primarily oxide or sulfide inclusions incorporating trace metallic impurities. These small inclusions were studied using primarily the Philips CM30 microscope. The probe size for this microscope tended to be larger than the precipitates, thus the primary element observed in each of the EDX spectra was matrix Fe. To remove the Fe signal from the spectra, a second spectrum, acquired from a region beside the precipitate, was subtracted from the first spectrum. Any element that was still visible in the resultant spectrum was listed in the tables. For the small oxide precipitates, it should be noted that while Fe is not explicitly listed in the tables due to the method described above, the weakness of the Cr and Mn signals in these modified spectra makes it almost impossible for the small oxides to not contain Fe.

The other main precipitate type observed in these alloys the authors have chosen to call "carbonitride flakes," meaning they are either ϵ -carbide platelets, α' -nitride platelets, or a combination of both. A discussion of attempts to distinguish between the two precipitate types follows this section on the observations. Higher densities of carbonitrides were observed in the alloys containing large amounts of nitrogen. For example, regions of VM349 (Fe-80 appm N) contained homogeneous distributions of carbonitride flakes with densities of $\sim 6.6 \times 10^{19} \text{ m}^{-3}$.

Each of the alloys will be described below. For each alloy there is a brief discussion of the pertinent observations and a standardized table in which these observations are summarized to allow easy comparisons between alloys. Each table is followed by the figures referred to in the discussion.

VM348: Fe-5 appm N (refer to Table 2)

This low-N alloy exhibited a duplex grain structure which resulted in slightly different defect structures in each of the regions. The defect microstructure in the large-grained region is shown in the low-magnification TEM micrograph displayed in Figure 2. Besides the low dislocation density visible in the image, the primary defects were the large carbonitride flakes which have heterogeneously precipitated on a low density of small Fe_3O_4 inclusions. A more complete study of these flakes appears in the sections on high-N alloys due to their much higher density in such alloys. The typical defect microstructure in the small-grained region of this low-N alloy is shown in the low-magnification TEM micrograph displayed in Figure 3. The primary defects in this region were carbonitrides which precipitated on the dislocations within the small grains. An example of such carbonitride formation is shown at higher magnification in Figure 4.

Large, spherical inclusions, like the one displayed in Figure 5, were observed in both the small- and large-grained regions. EDX analysis showed they contained Fe and O and tended to have a slight Cr enrichment. Diffraction analysis of the inclusions was consistent with the crystal structure of Fe_3O_4 . A low density of small intergranular oxides enriched with Cr was also observed in both regions of the alloys. In the large-grained region, they became the nucleation site for the carbonitride flakes seen in Figure 2. In the small-grained region, they generally showed little or no associated carbonitride formation as shown by Figure 6. These small oxides are believed to be Fe_3O_4 with strong Cr enrichment.

Table 2. Microstructural observations in VM348 (Fe-5 appm N)

Description	Size	Elements (Z > 11) major/minor	Density (m ⁻³)	Comments
Large, spherical inclusion	~ 1 μm	Fe / Cr		Fe ₃ O ₄ with slight Cr enrichment
Intergranular oxide	100 nm	Cr	1 x 10 ¹⁸	Believed to be Fe ₃ O ₄ with slight Cr enrichment
Associated carbide flake	~ 0.5 μm		1 x 10 ¹⁸	(1) On {100} planes ^a (2) Associated with each oxide

Notes: Observed duplex grain size, populations at ~ 10 μm and ~ 100 μm .

^aBelieved to be ϵ -carbide.

Figure 2. TEM micrograph showing the precipitate microstructure in a large-grained region of VM348; specimen thickness was 370 nm.

Figure 3. TEM micrograph showing precipitate microstructure in a small-grained region of the VM348 alloy.

Figure 4. TEM micrograph of carbonitride formation on a dislocation in the small-grained region of the VM348 alloy.

Figure 5. TEM micrograph of an Fe₃O₄ inclusion in VM348. The large, spherical inclusions tended to show slight Cr enrichment in this alloy.

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Figure 6. TEM micrograph of a Cr-enriched oxide in the small-grained region of the VM348 alloy. The oxides in this region showed very little tendency to form associated carbonitrides.

VM 349: Fe-80 appm N (refer to Table 3)

This alloy exhibited a higher density of carbonitride formation due primarily to the higher density of small Cr-enriched Fe_3O_4 intergranular oxides that acted as nucleation centers. As can be seen in the low-magnification image displayed in Figure 7, the flakes are, in general, slightly larger than those observed in the low-N alloy (see Figure 2). The image was acquired with the electron beam just off the $[100]_\alpha$ axis and clearly shows the carbonitride flakes which formed on the $\{100\}$ planes. When several of the small oxide precipitates formed close together, the associated flakes tended to be smaller, as can be seen in Figure 8. Figure 9 more clearly shows the imperfect $\{100\}$ planar habit of the carbonitride flakes which nucleated and grew on the $\{011\}$ faceted Cr-rich oxide. Large, spherical inclusions of slightly Cr-enriched Fe_3O_4 were also observed in this alloy.

In regions in which few oxide precipitates formed and were thus unavailable as nucleation sites, the carbonitride flakes precipitated homogeneously with a higher density, as can be seen in Figure 10. A couple of the small carbonitrides are shown at higher magnification in Figure 11.

Table 3. Microstructural observations in VM349 (Fe-80 appm N)

Description	Size	Elements (Z > 11) major/minor	Density (m^{-3})	Comments
Large, spherical inclusion	$\sim 1 \mu\text{m}$	Fe / Cr		Fe_3O_4 with slight Cr enrichment
Intergranular oxide	$\sim 50 \text{ nm}$	Cr	2.5×10^{18}	Believed to be Fe_3O_4 with slight Cr enrichment
Associated carbonitride flake	$\sim .5 \mu\text{m}$		2.5×10^{18}	(1) On $\{100\}$ planes ^a (2) Associated with each oxide
Homogeneous carbonitride flake	50 nm		6.6×10^{19}	(1) On $\{100\}$ planes (2) May be α' Fe_8N

Notes: Observed grain size was $\sim 100 \mu\text{m}$.

^aBelieved to be α' -nitride.

Figure 7. TEM micrograph showing the precipitate microstructure in the VM349 alloy. The image was taken with $g = 011$, near the $[100]_{\alpha}$ zone axis. Note the extensive carbonitride formation.

Figure 8. TEM micrograph showing the extensive carbonitride formation on intergranular oxides in the VM349 alloy.

Figure 9. TEM micrograph showing the $\{100\}$ planarity of the heterogeneous carbonitride precipitation on the Cr-enriched oxide, taken with $g = 011$ near the $[100]_c$ zone axis.

Figure 10. TEM micrograph of homogeneously precipitated carbonitride platelets in a region of the VM349 alloy in which very few oxides had precipitated.

Figure 11. TEM micrograph showing two of the carbonitride precipitates at higher magnification.

VM350: Fe-120 appm N (refer to Table 4)

This alloy exhibited a higher density of carbonitride formation than observed in the previous alloy (see Figure 7) due to the higher density of the small intergranular oxides that acted as nucleation sites. The carbonitrides tended to have a smaller diameter as seen in the low-magnification image displayed in Figure 12. This decrease in the size of the carbonitrides with an increase in the nucleation site density is most obvious in the lower right-hand corner of Figure 12 where the density of the oxides was about twice that in the rest of the image. A higher magnification image of a typical Cr-enriched intergranular oxide precipitate and its associated carbonitride flake are displayed in Figure 13. The inlayed picture is a higher magnification image of the oxide showing the characteristic parallel moiré fringe pattern typical of the oxides when they are encapsulated within the Fe matrix. Large, spherical inclusions of Cr-enriched Fe_3O_4 were also observed in this alloy. These inclusions tended to also show slight amounts of some impurities such as P and Si.

Table 4. Microstructural observations in VM350 (Fe-120 appm N)

Description	Size	Elements (Z > 11) major/minor	Density (m^{-3})	Comments
Large, spherical inclusion	$\sim 1 \mu\text{m}$	Fe / Cr		Fe_3O_4 with slight Cr enrichment; some contain P and Si
Intergranular oxide	50 nm	Cr	7×10^{18a}	Believed to be Fe_3O_4 with slight Cr enrichment
Associated carbonitride flake	$\sim .5 \mu\text{m}$		7×10^{18a}	(1) On {100} planes ^b (2) Associated with each oxide

Notes: Observed grain size was $\sim 100 \mu\text{m}$.

^aA small region was observed where the concentration of oxide precipitates was approximately twice that of the typical regions which resulted in smaller associated carbonitride flakes ($\sim 150 \text{ nm}$).

^bBelieved to be α' -nitride.

Figure 12. TEM micrograph showing the precipitate microstructure in the VM350 alloy. Specimen thickness was approximately 400 nm. Note the extensive carbonitride formation.

Figure 13. TEM micrograph of typical carbonitride formation on a small Cr-enriched oxide precipitate in the VM350 alloy. The inlay shows the oxide at a higher magnification displaying moiré fringes.

VM 390: Fe-0.5% Cu-20 appm N (refer to Table 5)

This alloy exhibited smaller than expected grain size ($\sim 10 \mu\text{m}$) although most of the grains were separated by low-angle grain boundaries. This small grain size is clearly visible in the low-magnification image displayed in Figure 14. Figure 15 shows two of the small Mn-enriched intergranular oxides that precipitated in this alloy, one with an associated carbonitride flake clearly visible in the center of the image, and one without in the lower left-hand corner of the image. Less than half of the oxides nucleated carbonitride flakes. The carbonitrides nucleated preferentially on dislocations, one of which also appears in Figure 15. This observation is similar to the small-grained region of VM348 (see Figures 4 and 6). Large, spherical inclusions of Mn-enriched Fe_3O_4 were also observed in this alloy.

Table 5. Microstructural observations in VM390 (Fe-0.5% Cu-20 appm N)

Description	Size	Elements (Z > 11) major/minor	Density (m^{-3})	Comments
Large, spherical inclusion	$\sim 1 \mu\text{m}$			
Intergranular oxide	50 nm	Mn	$\sim 7 \times 10^{18}$	Believed to be Fe_3O_4 with slight Mn enrichment
Associated carbonitride flake	150 nm		$<< 7 \times 10^{18}$	C/ N preferentially precipitated on dislocations ^a

Notes: Observed grain size was $\sim 10 \mu\text{m}$. Most grain boundaries observed were low-angle grain boundaries.

^a Believed to be α' -nitride.

Figure 14. TEM micrograph showing the precipitate microstructure in the VM390 alloy. Note the smaller-than-expected grain size. There were many low-angle grain boundaries observed.

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Figure 15. TEM micrograph of Mn-enriched oxide and carbonitride formation (preferentially on dislocations) observed in the VM390 alloy. The oxide in the lower left of the image showed no associated carbonitride formation.

VM397: Fe-0.9% Cu-20 appm N (refer to Table 6)

This alloy was the first to show virtually no carbonitride formation, as is evident by the lack of any visible carbonitride flakes in the low-magnification image displayed in Figure 16. Also visible in the figure are small intergranular precipitates that were observed to be aligned along what are assumed to be prior grain boundaries, either from austenite grain boundaries formed during the solution treatment, or from grain boundaries present before the solution treatment. Unlike the intergranular oxides observed in the previous alloys, these precipitates were observed to be two phase, as can clearly be seen in Figure 17. Figure 17(a) shows two small, round precipitates of which the rightmost one shows a strongly diffracting faceted inner phase. Figure 17(b) shows a larger round precipitate with the inner phase displaying a moiré fringe pattern while the outer phase appears amorphous. These small precipitates generally showed either Mn, or Mn and Cr enrichment, but their crystal structure was not determined. The large, spherical inclusions observed in this alloy also deviated from those in previous alloys. Figure 18 shows a typical inclusion for this alloy, with A marking the Mn-enriched Fe_3O_4 , and B marking an associated precipitate believed to be an iron silicide, which also appears to be Mn enriched.

Table 6. Microstructural observations in VM397 (Fe-0.9% Cu-20 appm N)

Description	Size	Elements (Z > 11) major/minor	Density (m^{-3})	Comments
Large, spherical inclusion	$\sim 2 \mu\text{m}$	Fe, Mn/Cr		Fe_3O_4 with slight Mn enrichment
Associated with inclusion	$< .5 \mu\text{m}$	Si, Mn		Probably Mn-enriched iron silicide
Intergranular precipitates	$< 50 \text{ nm}$	Mn and Mn, Cr	low $< 1 \times 10^{18}$	(Mn/Cr) > 1 Images showed two-phase precipitates ^a

Notes: Observed grain size was $> 100 \mu\text{m}$. There was no apparent carbonitride formation in this alloy.

^aThe intergranular precipitates appear mainly along what are assumed to be prior grain boundaries.

Figure 16. TEM micrograph showing the precipitate microstructure in the VM397 alloy. Note that the observable precipitates appear to follow prior grain boundaries, and note the complete lack of any carbonitride formation.

Figure 17. TEM micrographs showing the two-phase nature of the precipitates observed in the VM397 alloy. In (a) the central phase shows strong diffraction, and in (b) the central phase shows moiré fringes while the outer phase appears amorphous.

Figure 18. TEM micrograph of a typical large inclusion in the VM397 alloy. The part of the inclusion marked A is Fe_3O_4 with slight Mn enrichment, and the part marked B is believed to be an Mn-enriched iron silicide.

VM399: Fe-0.5% Cu-120 appm N (refer to Table 7)

This alloy exhibited a microstructure very similar to that of the VM 349 alloy (Fe + 80 appm N) as can be seen by comparing the low-magnification image for this alloy, Figure 19, with the corresponding one for the VM349 (see Figures 7 and 8). The two alloys have comparable densities of the small Cr-enriched intergranular oxides, believed to be Fe_3O_4 , as well as similarly sized associated carbonitride flakes. A high-magnification image of one of the smaller flakes edge-on showing the discontinuous nature of the carbonitride flake, and the small Cr-rich precipitate on which it nucleated appears in Figure 20. Large, spherical inclusions of Cr-enriched Fe_3O_4 were also observed in this alloy.

Table 7. Microstructural observations in VM399 (Fe-0.5% Cu-120 appm N)

Description	Size	Elements (Z > 11) major/minor	Density (m^{-3})	Comments
Large, spherical inclusion	$\sim 1 \mu\text{m}$			Fe_3O_4 Cr and Mn enrichment
Intergranular oxide	$\sim 50 \text{ nm}$	Cr	2×10^{18}	(Cr / Mn) > 1 Believed to be Fe_3O_4
Associated carbonitride flake	$\sim 1 \mu\text{m}$		2×10^{18}	(1) On {100} planes ^a (2) Associated with each oxide

Notes: Observed grain size was $> 100 \mu\text{m}$.

^aBelieved to be α' -nitride.

Figure 19. TEM micrograph showing the precipitate microstructure in the VM399 alloy. Note the extensive carbonitride formation.

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Figure 20. TEM micrograph of a carbonitride flake edge-on. Note the discontinuous nature of the carbonitride which precipitated on the Cr-enriched oxide.

VM360: Fe-0.9% Cu-1% Mn-10 appm N (refer to Table 8)

This alloy exhibited the widest range of precipitate sizes of all the alloys. The very large cylindrical inclusions, like those shown in Figure 21, had diameters up to 1 μm and lengths of up to 25 μm . EDX showed these inclusions to contain Mn and O, and diffraction analysis showed their crystal structure to match that of MnO. Figure 22 shows one of the stringers partially exposed from the matrix. At the top left edge of the inclusion is an associated precipitate which EDX showed to be Cu_xS . A low density of small precipitates was observed throughout the alloy, as shown in Figure 23. EDX revealed that these precipitates contained Mn and S, and diffraction analysis showed them to have a cubic structure consistent with α MnS. The evidence for a set of even smaller precipitates is displayed in Figure 24. The images displayed in this figure are of different dislocations at various stages of moving past unobserved barriers in this alloy. Figure 24(a) shows a dislocation just as it begins to be pinned at a barrier, (b) shows a dislocation curved about a barrier as it moves past it, (c) shows a dislocation just as it cuts off the pinned portion of the dislocation, and (d) shows a dislocation which has moved past a barrier leaving behind a small dislocation loop. This behavior strongly suggests that the unobserved barriers were incoherent with the matrix.

Table 8. Microstructural observations in VM360 (Fe-0.9% Cu-1% Mn-10 appm N)

Description	Size	Elements (Z > 11) major/minor	Density (m^{-3})	Comments
Large, cylindrical inclusion	$D \leq 1 \mu\text{m}$ Length up to 25 μm	Mn		Bamboo structure Appears to be MnO
Associated with inclusions	250 nm	Cu, S		Believed to be Cu_xS
Intergranular sulfide	$\sim 50 \text{ nm}$	Mn, S	very low $<< 1 \times 10^{18}$	α MnS
Unknown precipitate	$< 10 \text{ nm}$			^a

Notes: Observed grain size was $\sim 50 \mu\text{m}$. There was no apparent carbonitride formation in this alloy.

^aThe evidence for these precipitates is the pinning of dislocations. The formation of dislocation loops about the precipitates instead of shearing strongly suggests the precipitates are incoherent with the matrix.

Figure 21. TEM micrograph showing the precipitate microstructure in the VM360 alloy. Note the long MnO stringers displaying a bamboo structure.

Figure 23. TEM micrograph showing a typical α MnS precipitate observed in the VM360 alloy. A low density of these precipitates was observed throughout the specimen.

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Figure 22. TEM micrograph showing a smaller inclusion and an associated precipitate which is believed to be a copper sulfide.

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Figure 24. TEM micrographs showing different dislocations at various stages of moving past small, unobserved precipitates: (a) shows a dislocation which has just made contact with an unobserved precipitate, (b) shows a dislocation as it bends around a precipitate, (c) shows a dislocation pinching off as it passes a precipitate, and (d) shows a pinched-off dislocation loop pinned by several small precipitates and the dislocation which has moved past the precipitates.

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VM387: Fe-0.5% Cu-0.17% C-10 appm N (refer to Table 9)

This alloy exhibited surprisingly little precipitation in its microstructure, as can be seen in the low-magnification image displayed in Figure 25. Several large precipitates believed to be cementite particles were observed. The small density of carbonitride flakes that did form was observed to have nucleated on small TiC precipitates, as shown in Figure 26. The small carbonitride flake nucleated on this precipitate was etched away during the polishing process.

Table 9. Microstructural observations in VM387 (Fe-0.5% Cu-0.17% C-10 appm N)

Description	Size	Elements (Z > 11) major/minor	Density (m ⁻³)	Comments
Large, spherical inclusion	~ 1 μm			
Intergranular carbide	~ 20 nm	Ti	3.5×10^{18}	Believed to be TiC
Associated carbon flake	~ .25 μm		3.5×10^{18}	Believed to be ϵ -carbide
Cementite	$\leq 2 \mu\text{m}$			Not on {100} planes

Notes: Observed grain size was > 100 μm .

Figure 25. TEM micrograph showing the precipitate microstructure in the VM387 alloy. There was surprisingly little precipitation in this high-carbon alloy.

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Figure 26. TEM micrograph of a typical TiC precipitate on which the small carbonitrides (preferentially etched during the polishing process) formed.

VM373: Fe-0.3% Ti-100 appm N (refer to Table 10)

This alloy was the only high-N alloy that did not exhibit extensive carbonitride formation. Figure 27 displays a low-magnification image of the microstructure observed in this alloy. TiC precipitates were observed in this alloy with a wide size distribution. They were observed to be as large as 2 μm , and as small as 100 nm, such as the one particle that is displayed in Figure 28. They are recognizable by their tendency to facet along the $\{100\}_\alpha$ planes and display strong moiré fringing under certain diffraction conditions. A set of small precipitates formed along what appear to be prior grain boundaries, similar to the pattern observed in the VM397 alloy (see Figure 16). EDX analysis of the precipitates that appear in Figure 29 shows that they contain Ti and S. Another set of small intergranular precipitates was observed to give rise to strong moiré fringes under certain conditions, similar to the precipitate displayed in Figure 30, but showed no heavy elements ($Z > 11$) other than Fe. It is possible that these precipitates are the small Fe_3O_4 inclusions observed so often in the other alloys but without the Cr or Mn enrichment.

Table 10. Microstructural observations in VM373 (Fe-0.3% Ti-100 appm N)

Description	Size	Elements ($Z > 11$) major/minor	Density (m^{-3})	Comments
Large, rectangular precipitates	$\leq 2 \mu\text{m}$	Ti		TiC Partially coherent with matrix
Intergranular sulfide	$\leq 50 \text{ nm}$	Ti / S	$\sim 2 \times 10^{19a}$	Follow old grain boundary Believed to be Ti_xS
Intergranular precipitate	$\sim 25 \text{ nm}$		$\sim 2 \times 10^{19a}$	Possibly Fe_3O_4

Notes: Observed grain size was $\sim 50 \mu\text{m}$. There was no apparent carbonitride formation in this alloy.

^aThis value includes both the sulfides and the unidentified precipitates.

Figure 27. TEM micrograph showing the precipitate microstructure in the VM373 alloy. Note the lack of carbonitride formation in this high-Ti-high-N alloy.

Figure 28. TEM micrograph showing a typical TiC particle which precipitated in the VM373 alloy with the arrow marking the $g = 011$ reflection. The TiC particles were as large as $2\ \mu\text{m}$, and all were strongly faceted.

Figure 29. TEM micrograph showing unfaceted precipitates that formed along prior grain boundaries in the VM373 alloy. The precipitates contain Ti and S.

Figure 30. TEM micrograph showing one of a set of small precipitates which showed strong moiré fringing but did not register any heavy elements other than Fe.

Determination of Precipitate Character; Carbide or Nitride

An attempt to determine if the carbonitride flakes were ϵ -carbide flakes or α' -nitride flakes using imaging techniques was undertaken in two of the alloys, the alloy with the highest N content, VM350, and the alloy with the highest C content, VM387. Normally, both the carbide and the nitride precipitate as platelets on the $\{100\}$ planes and are capable of being nucleated on dislocations making differentiation difficult.

The precipitates are reported to be distinguishable by the shape of the precipitate edge when viewed flat-on;⁶ the α' -nitrides have smooth edges, and the ϵ -carbides have ragged edges and a "flower-like" appearance. Using this criterion, all the large carbonitride flakes would seem to be ϵ -carbides, even in the high-N alloys. A more recent study than the one cited above seems to suggest that the α' -nitrides also display "rosette" contrast when viewed flat-on, and although the edges seem somewhat smooth, at least one of their images shows what looks like an α' -nitride with a ragged edge.⁷ This would seem to bring the conclusion that all the carbonitrides were ϵ -carbides into question.

Another method suggested by the original study,⁶ and used successfully in studies of alloys similar to the ones studied here,^{8,9} distinguishes between the precipitates by using the contrast of the precipitates in images acquired with different diffracting conditions. It uses the fact that the α' -nitrides induce strains in the iron matrix in the $\langle 100 \rangle$ direction perpendicular to the habit plane, while the ϵ -carbides induce strain in one of the lateral directions as well as the thickness direction. Therefore, under the appropriate $\langle 200 \rangle$ diffraction condition, α' -nitrides should show only residual contrast while the ϵ -carbides show much stronger contrast.

An example of this technique applied to the high-N alloy VM350 appears in Figure 31. The figure shows the same carbonitride platelets taken under several different diffracting conditions, marked by the arrow in each image, all with the foil tilted just off the $[100]_{\alpha}$ axis. Figures 31(a) and (b) were acquired with $g = 011$ and $g = 0\bar{1}1$, diffracting conditions, respectively. Under these conditions both of the precipitate types should be, and are, clearly visible. Figure 31(c) was acquired with the $g = 0\bar{2}0$ diffracting condition. Under this condition the contrast from the α' -nitrides with habit planes on the (001) [i.e., lying along the arrow direction] should be reduced to residual contrast while the contrast from ϵ -carbides should remain fairly strong. The top section of the crossed precipitate at the center, along with a few of the smaller platelets on the dislocation directly above the arrow and on dislocations on the right side of the image, show very little contrast, suggesting they are nitrides. The lower section of the crossed precipitate at the center, along with the precipitate in the lower right-hand side of the images, still shows strong contrast, suggesting they are carbides. Figure 31(d) was acquired with the $g = 002$ diffracting condition. Under this condition the contrast from the α' -nitrides with habit planes on the (010) [i.e., lying along the arrow direction] should be reduced to residual contrast while the contrast from ϵ -carbides should remain fairly strong. The middle section of the crossed precipitate at the center, along with a few of the smaller platelets on the dislocation directly above the arrow and to the right side of the image, shows very little contrast, suggesting they are nitrides. The results of this contrast investigation, although a bit ambiguous, suggest that in the high-N alloys the small platelets associated with dislocations are likely α' -nitrides, while the larger flakes that precipitated on the intergranular oxides may be a combination of ϵ -carbides and α' -nitrides. A preliminary study employing aperture displaced dark field imaging supports the hypothesis that most of the carbonitride flakes in the high-N alloys are α' -nitrides.

An example of the technique applied to the high-C alloy VM387 appears in Figure 32. The figure shows the same carbonitride platelets taken under several different diffracting conditions, marked by the arrow in each image, all with the foil tilted just off the $[100]_{\alpha}$ axis. Figures 32(a) and (b) were acquired with the $g = 011$ diffracting condition. All the precipitates show strong contrast, including the long particle in Figure 32(b), which is believed to be a cementite precipitate. Figures 32(c) and (d) were acquired with the $g = 00\bar{2}$, diffracting condition. The platelets with habit planes lying along the arrow direction only show slightly reduced contrast suggesting that they are ϵ -carbides. Figures 32(e) and (f) were acquired with the $g = 020$ diffraction condition. The horizontal platelets in (e) and (f) again show reduced contrast but not really sufficient to suggest they are nitrides. This image contrast investigation suggests that, in the high-C alloy, all the flakes that precipitated are ϵ -carbides.

Figure 31. TEM micrographs of the same {100} platelets acquired under different diffracting conditions with the electron beam just off the $[100]_g$ axis: (a) $g = 011$, (b) $g = 0\bar{1}1$, (c) $g = 0\bar{2}0$, and (d) $g = 002$.

Figure 32. TEM micrographs of the same precipitates acquired under different diffracting conditions with the electron beam just off the $[100]_a$ axis: (a) and (b) were taken with $g = 011$, (c) and (d) with $g = 002$, and (e) and (f) with $g = 020$.

Discussion of Observed Solute Effects

Although many of the solutes were present only at the level of impurities, some conclusions can be drawn about the effects of the various alloying elements on the observed microstructure by comparing the results of this study in a systematic fashion. By comparing the microstructures of VM348, VM349, and VM350, it is possible to say that one of the major effects of increasing the N content was to increase the intergranular oxide formation and its associated carbonitride formation significantly. A comparison of the microstructures of VM390 and VM399 indicates that the increased N content in the Cu-containing alloys increased the ratio of Cr to Mn which was incorporated into the oxide formation.

It is possible to follow a similar series of alloys to investigate the effects of increased Cu content on the microstructure of these alloys. When comparing VM348, VM390, and VM397, the only consistent effect observed in these low-N alloys seems to be the increased ratio of Mn to Cr incorporated into the oxide formation. Most other insights are lost to inconsistencies, such as the apparent increase in oxide formation for the 0.5% Cu alloy but reduction in oxide formation in the 0.9% Cu alloy. Perhaps a better comparison for the increase in Cu content is the high-N alloys VM349 and VM399. Their microstructures were remarkably similar, showing no significant change in oxide formation or carbonitride formation, except that no homogeneous precipitation of the carbonitride flakes was observed in the Cu-containing alloy. Given the fact that VM399 has a higher N content and a lower density of nucleation sites (i.e., oxide precipitates) than VM349, this lack of homogeneous precipitation may mean that the driving force for carbonitride formation is reduced in the Cu-containing alloys. This would be supported by the lack of carbonitride formation in the highest Cu-containing alloy, VM397.

Comparisons of other alloys are possible, but they are less systematic, and thus there is less confidence placed in the conclusions that are drawn from them. By comparing VM397 with VM360, we can note effects due to increased Mn content in the alloys. There seems to be an increase in sulfide precipitation, both intergranular and associated with the inclusions, when Mn is added. As well, the O was incorporated into the cylindrical inclusions, and thus there was no small intergranular oxide formation observed. By comparing VM387 with VM390, we can note the effects due to the increased C content in the alloys. The most obvious effect is the increase in cementite (Fe_3C) formation, which was only rarely seen in the other alloys. The intergranular oxide formation was replaced by TiC precipitation, and, surprisingly, the carbonitride formation seemed to be slightly reduced. By comparing VM373 with VM350, it is possible to note the effects due to increased Ti content in the alloys. Most notably, the addition of Ti was observed to eliminate the carbonitride formation. Less significantly, the oxide inclusions were replaced with large TiC particles, and, again, some sulfides were observed.

Summary

This TEM characterization of the model alloys in their as-received condition will provide a baseline for comparison with specimens that are being ion irradiated. The results have confirmed that the alloys are reasonably clean and appropriate for their intended use. Grain sizes were generally large, $\sim 100\ \mu\text{m}$, and the dislocation densities were quite low, $\sim 10^{13}\ \text{m}^{-2}$. The total precipitate density was also low, on the order of 1 to $7 \times 10^{18}\ \text{m}^{-3}$. The precipitates were primarily oxide or sulfide inclusions incorporating trace metallic impurities. Higher densities of carbonitrides were observed in the alloys containing large amounts of nitrogen.

Although some interesting effects of impurities were observed, the alloying elements Cu, C, N, and Ti that were intentionally added appeared to have had the desired effects. Some evidence of copper precipitation during the initial heat treatment was found by comparing the hardness of thermally aged material with similar material that had received a somewhat different heat treatment at the AEA Harwell Laboratory. This difference is not significant to the ion irradiation experiments that are currently under way.

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2. TITLE AND SUBTITLE Microstructural Characterization of Selected AEA/UCSB Model FeCuMn Alloys		3. DATE REPORT PUBLISHED <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; text-align: center;">MONTH</td> <td style="width: 50%; text-align: center;">YEAR</td> </tr> <tr> <td style="text-align: center;">June</td> <td style="text-align: center;">1996</td> </tr> </table>	MONTH	YEAR	June	1996
MONTH	YEAR					
June	1996					
5. AUTHOR(S) P. M. Rice and R. E. Stoller		4. FIN OR GRANT NUMBER L1098				
8. PERFORMING ORGANIZATION -- NAME AND ADDRESS <i>(If NRC, provide Division, Office or Region, U.S. Nuclear Regulatory Commission, and mailing address; if contractor, provide name and mailing address.)</i> Oak Ridge National Laboratory Oak Ridge, TN 37831-6285		6. TYPE OF REPORT Technical				
9. SPONSORING ORGANIZATION -- NAME AND ADDRESS <i>(If NRC, type "Same as above"; if contractor, provide NRC Division, Office or Region, U.S. Nuclear Regulatory Commission, and mailing address.)</i> Division of Engineering Technology Office of Nuclear Regulatory Research U.S. Nuclear Regulatory Commission Washington, DC 20555-0001		7. PERIOD COVERED <i>(Inclusive Dates)</i> 				
10. SUPPLEMENTARY NOTES M. Vassilaros, NRC Project Manager						
11. ABSTRACT <i>(200 words or less)</i> A set of 22 model ferritic alloys was purchased as part of a collaborative research program by the AEA Harwell Laboratory and the University of California at Santa Barbara. Nine of these alloys were selected by the Oak Ridge National Laboratory for use in a series of ion irradiation experiments investigating dispersed barrier hardening. These nine alloys contain varying amounts of copper, manganese, titanium, carbon, and nitrogen. The alloys have been characterized by transmission electron microscopy in the as-received condition to provide a baseline for comparison with the irradiated specimens. A description of the microstructural observations is provided for future reference. This summary focuses on the type and size distributions of the precipitates present; grain size and dislocation measurements are also included.						
12. KEY WORDS/DESCRIPTORS <i>(List words or phrases that will assist researchers in locating the report.)</i> Microstructural Characterization Precipitates Pressure Vessel Steels Hardening Irradiation		13. AVAILABILITY STATEMENT Unclassified 14. SECURITY CLASSIFICATION <i>(This Page)</i> Unclassified <i>(This Report)</i> Unclassified				
		15. NUMBER OF PAGES 				
		16. PRICE 				