

# Influence of Long-Term Thermal Aging on the Microstructural Evolution of Nuclear Reactor Pressure Vessel Materials

## An Atom Probe Study

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Manuscript Completed: December 1997

Date Published: March 1998

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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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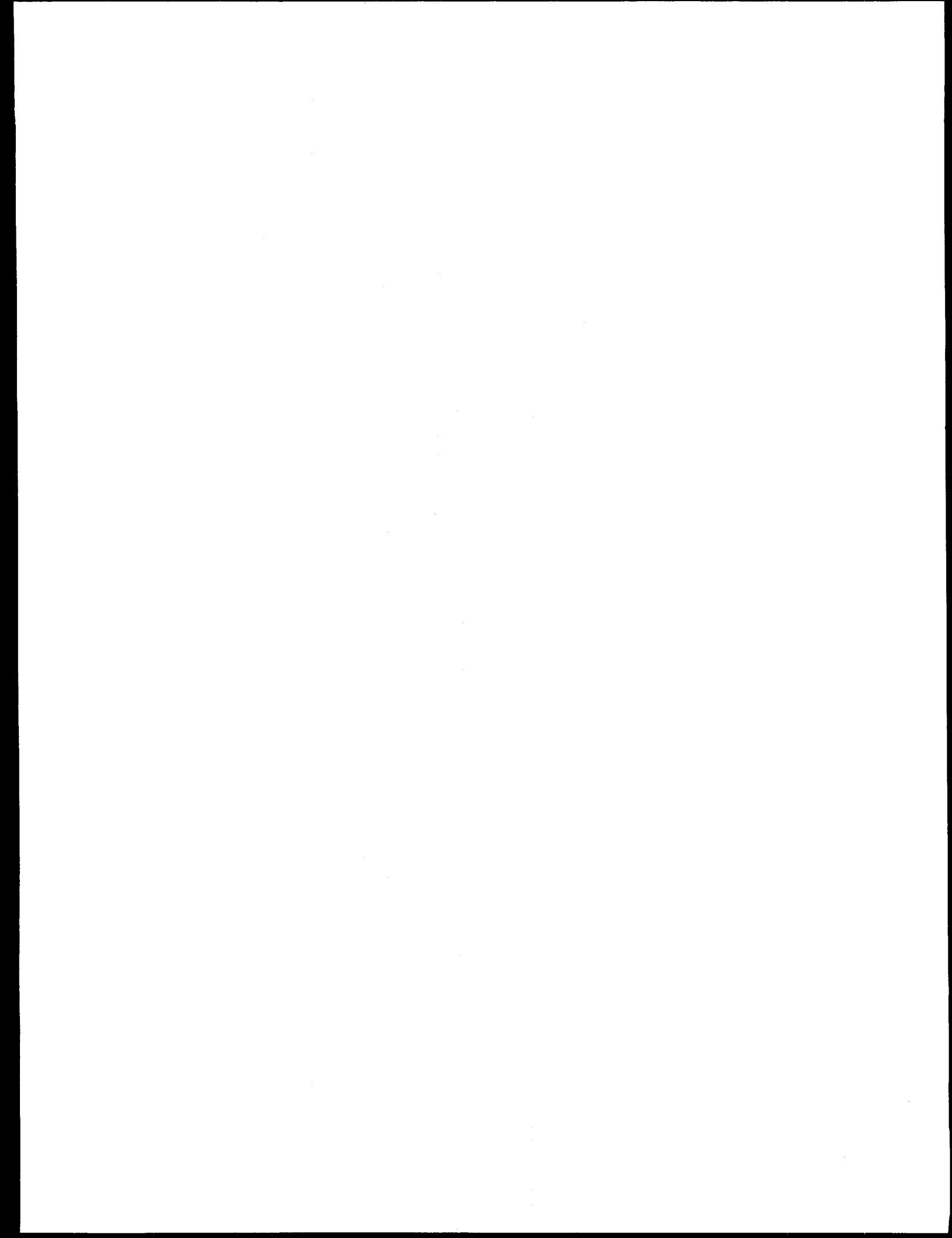
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## Abstract

Atom probe field ion microscopy (APFIM) investigations of the microstructure of unaged (as-fabricated) and long-term thermally aged (~100,000 h at 280°C) surveillance materials from commercial reactor pressure vessel steels were performed. This combination of materials and conditions permitted the investigation of potential thermal-aging effects. This microstructural study focused on the quantification of the compositions of the matrix and carbides. The APFIM results indicate that there was no significant microstructural evolution after a long-term thermal exposure in weld, plate, or forging materials. The matrix depletion of copper that was observed in weld materials was consistent with the copper concentration in the matrix after the stress-relief heat treatment. The compositions of cementite carbides aged for 100,000 h were compared with the Thermocalc™ prediction. The APFIM comparisons of materials under these conditions are consistent with the measured change in mechanical properties such as the Charpy transition temperature.

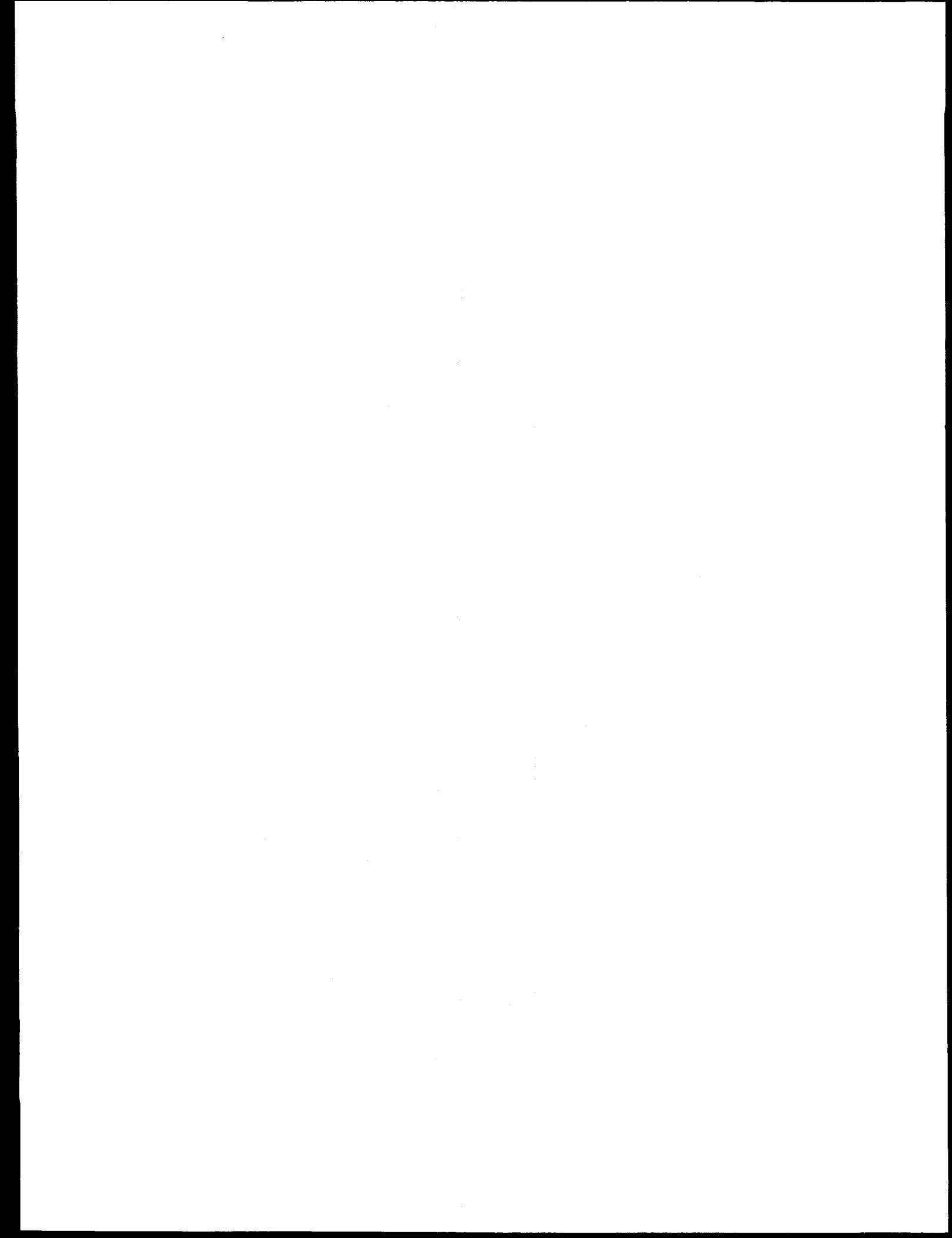
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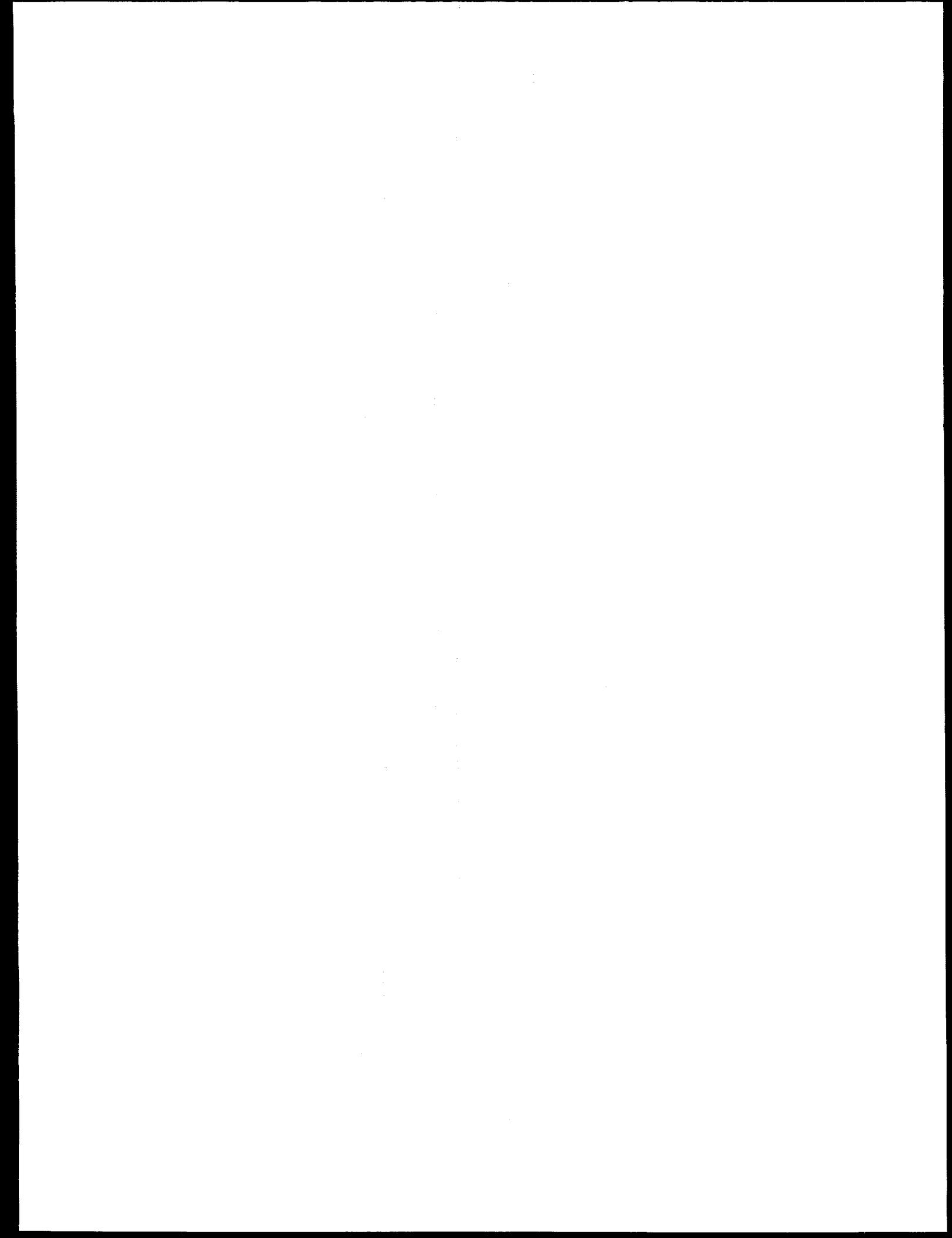
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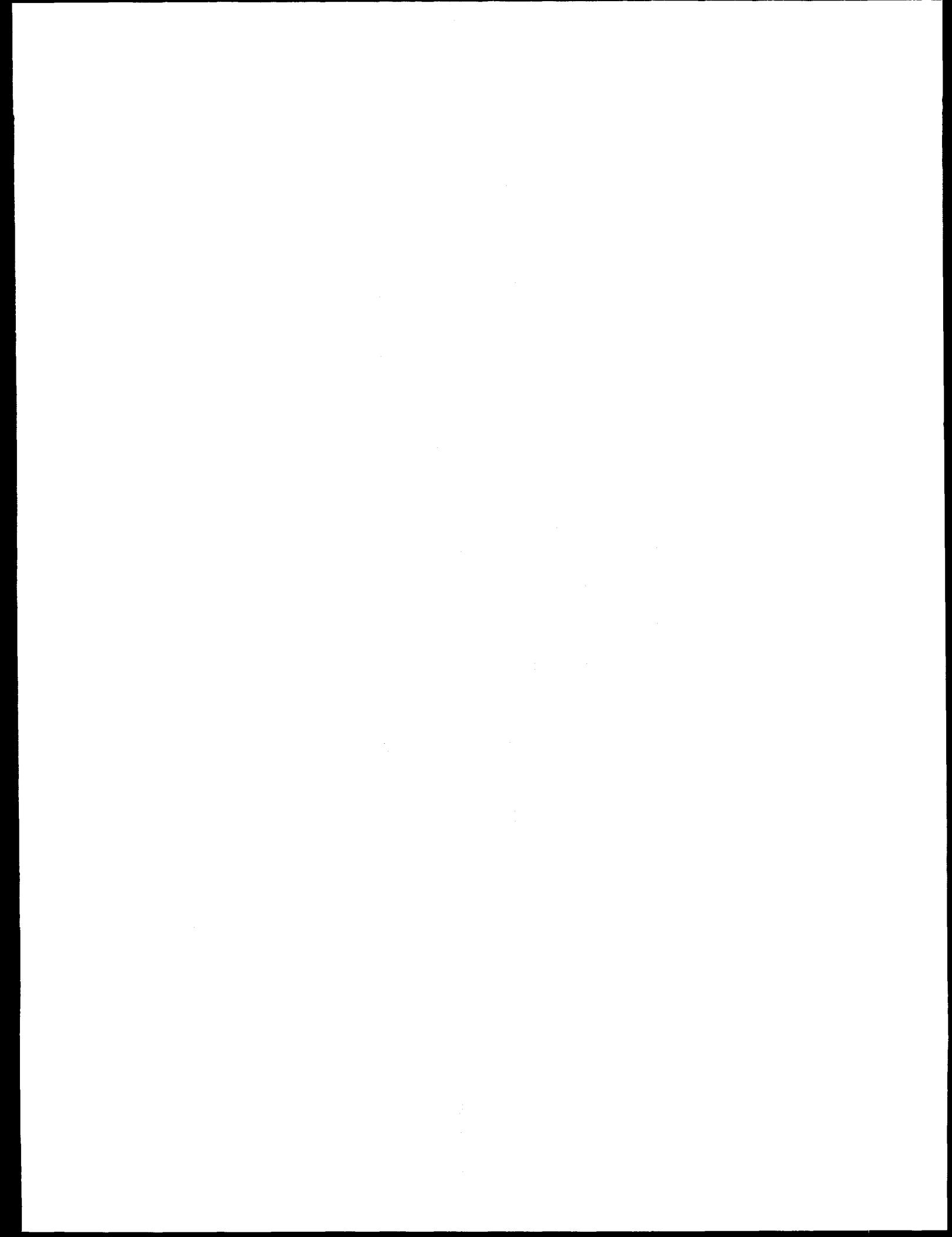
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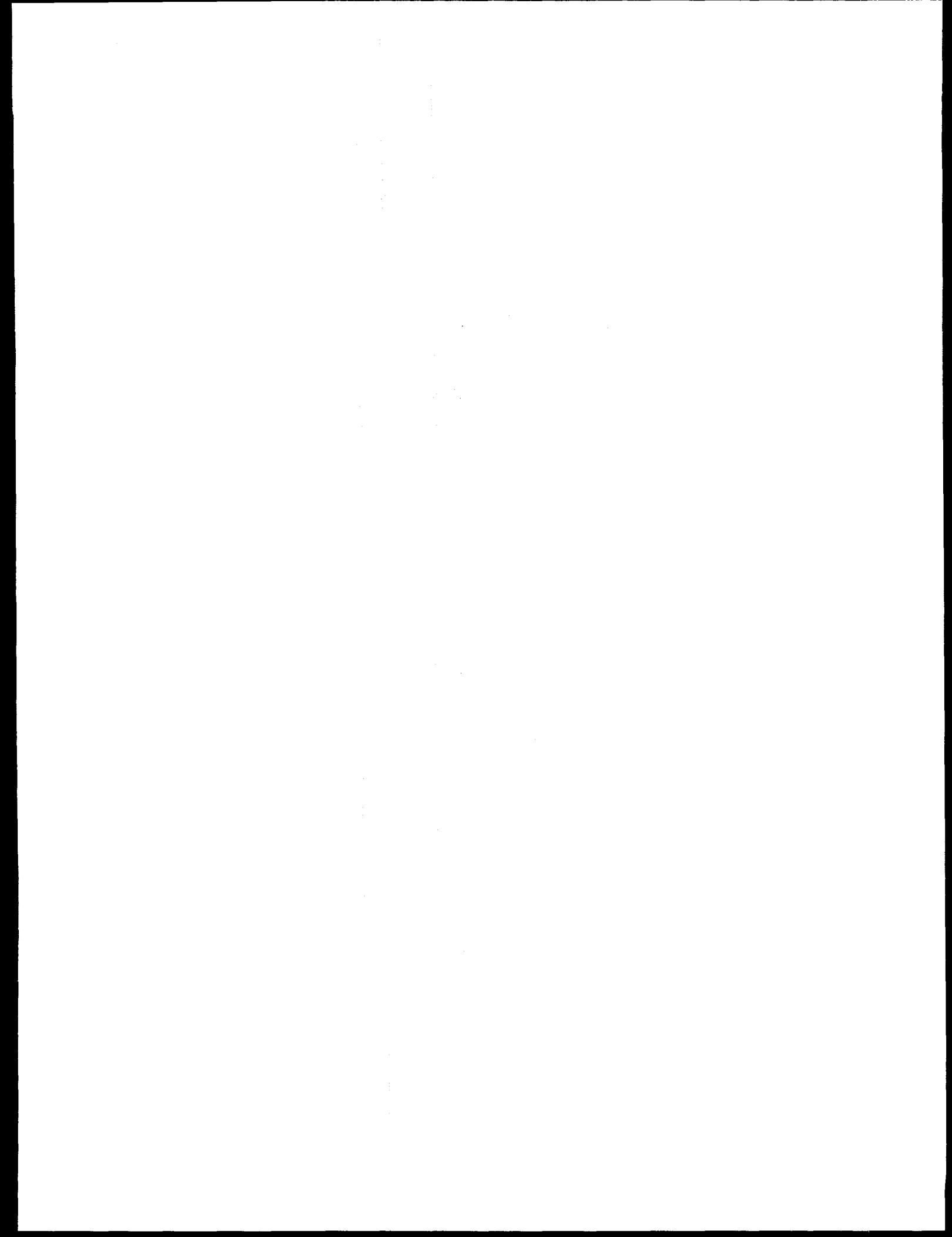
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## **Acknowledgments**

The materials for this study were provided by the Babcox and Wilcox (B&W) Owners Group. The authors would like to thank M. J. De Van and W. A. Pavinich of B&W Nuclear Technologies (now Framatome Technologies, Inc.) for their assistance. The continuing financial support of the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission, is gratefully acknowledged.

Contributions to this research were sponsored in part by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. and by the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission under interagency agreement DOE 1886-8109-8L with the U.S. Department of Energy. P. Pareige was supported by an appointment 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. This research was conducted utilizing the Shared Research Equipment (SHaRE) User Program facilities at Oak Ridge National Laboratory.



## 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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# Influence of Long-Term Thermal Aging on the Microstructural Evolution of Nuclear Reactor Pressure Vessel Materials: An Atom Probe Study\*

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## Introduction

Surveillance programs for nuclear reactor vessels were primarily designed to monitor the radiation-induced changes occurring in the mechanical properties of the pressure vessel materials. Nowadays, a large set of data is available on the mechanical properties and also the microstructural evolution of irradiated materials. The embrittlement of vessel steels and weldments is known to be related to the presence of residual elements such as copper and phosphorus. Recent microstructural investigations have conclusively shown that the presence of these elements leads to the radiation-enhanced or radiation-induced formation of ultrafine copper-enriched clusters associated with nickel, manganese, silicon, phosphorous, and iron.<sup>1-10</sup> However, this evolution of the microstructure has been observed in materials that have been neutron-irradiated for several years at temperatures near 290°C. It is therefore possible that some of the degradation in mechanical properties may be a result of the long-term thermal-aging component.

Some previous research has indicated the potential for thermal degradation of the mechanical properties of materials used in the construction of nuclear power systems when they are operated at relatively high temperatures (>371°C).<sup>11</sup> However, degradation in properties as a result of long-term exposure at lower temperatures (~300°C) is still an open question.<sup>12</sup>

Among the studies on pressure vessel steels, Pense<sup>13</sup> detected no shift in the ductile-to-brittle transition temperature (DBTT) of an A302 Mn-Mo plate steel after a relatively short aging time of 500 h at 370°C. After the same aging time, an A203 Mn-Ni steel exhibited shifts in the transition temperature in excess of 40°C. Thermal-aging data are also available for a SA-302B steel for periods of 9,726 h and 26,114 h at 307°C; these data were reported from the surveillance program of the Big Rock Point Reactor (BRP).<sup>14,15</sup> These data indicated little effect of thermal aging on the Charpy impact results of both the SA-302, Grade B modified steel (which is equivalent to the current SA-533, Grade B1 steel) base metal and weld metal. Also, one capsule from the Oconee Unit 1 pressurized water reactor (PWR) aged at 304°C for 15,800 h showed no significant shift in DBTT for the same materials as described above.<sup>16</sup> Recent results<sup>17</sup> concerning materials removed from Oconee Unit 3, aged for 103,000 h at 280°C, and from Arkansas Unit 1, aged for 93,000 h at 280°C, have shown that thermal aging had only a minor effect on the impact properties of both SA-302B base and weld metal. Both modest increases and decreases in the DBTT and the energy on the upper shelf were observed; however, the overall changes resulting from thermal aging were of such small magnitude as to be considered insignificant. Because the evaluation of the Charpy impact properties was inconclusive, the authors of Ref. 17 recommended that additional investigation should be performed to determine whether thermal aging was having an impact on these materials. They suggested that tensile and fracture toughness testing should be performed and that microstructural characterization of the materials was needed.

\*Research sponsored by the Office of Nuclear Regulatory Research, Division of Engineering Technology, U.S. Nuclear Regulatory Commission, under Interagency Agreement DOE 1886-8109-8L with the U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

The main objective of the work presented in this paper was to use the technique of atom probe field ion microscopy (APFIM)<sup>18,19</sup> to characterize the microstructure and the composition of low-temperature (~300°C), long-term (~100,000 h) thermally aged plate, forging, and weld materials from the Babcock and Wilcox (B&W) Master Integrated Reactor Vessel Surveillance Program in order to investigate the potential thermal-aging effects. It is important to determine whether there are any changes in the composition of the matrix and whether any ultrafine precipitates had formed due to the thermal component of the service environment only. If such changes were observed they would provide an early indication of the potential for thermal embrittlements at the longer times (~300,000 h) associated with the vessel lifetime.

Also, the embrittlement of pressure vessel steels and weldments during neutron irradiation is known to be related to the presence of residual elements, especially copper and phosphorus. Thus, it is of prime interest to have accurate estimates of the levels of these impurities in the materials prior to irradiation. These parameters are key factors in the prediction of the embrittlement of a pressure vessel during neutron irradiation, and they can only be measured by an ultrafine scale microstructural technique such as APFIM. Thus unaged specimens have been investigated with the atom probe in addition to characterizing the thermally aged materials.

## Materials Description

Five structural steels were selected for the examination of long-term thermal-aging effects. Both Oconee Unit 3 and Arkansas Unit 1 reactors have thermal-aging boxes containing pieces of surveillance materials. The material removed from the thermal-aging boxes included forging, plate, and two weld metals. The base and weld metals are representative of the materials used to fabricate the beltline shell course regions of the Oconee Unit 3 and Arkansas Unit 1 reactor pressure vessels. In addition, the boxes contained ASTM correlation monitor plate material. The base metal materials were SA-533, Grade B, Class 1 plate steel and SA-508, Class 2 forging steel. The two weld metals were typical Mn-Mo-Ni weld wire, Linde 80 flux submerged-arc welds. The chemical compositions of the five materials are reported in Table 1.

The specimen-aging capsules were located in boxes used for irradiation on the service structure support of the reactor vessel heads. The boxes were located under the head insulation to help maintain the aging temperature and within the flow of the entering coolant, which helped maintain the capsules at the same temperature as the reactor vessel wall. As a result of the location of these boxes, the surveillance material was exposed to an actual neutron fluence of less than  $1 \times 10^{14} \text{ n.m}^{-2}$ , or essentially zero insofar as material damage is concerned. The exact exposure time for each set of materials was difficult to determine because of the time allowance for reactor heat-up, cool-down, and hot standby. The aging times given in Table 2 for these materials reflect a 10% increase of the actual effective full-power times when the surveillance material was removed.

The materials have also been characterized in the unaged condition to have a full characterization of the microchemistry of materials prior to irradiation and to be able to determine the effects of exposure to temperature alone. The heat-treatment and stress-relief histories of these materials are given in Table 2.

## Experimental

The APFIM technique is particularly well suited to the characterization of these pressure vessel steels because of its near-atomic resolution and its ability to chemically analyze features on the near-atomic

Table 1. Chemical composition (bulk chemistry) of Babcox and Wilcox Owners Group surveillance materials

Identification	%	Cu	Ni	Mn	Si	P	C	S	Mo	Cr
Plate A	wt %	0.15	0.52	1.32	0.2	0.01	0.21	0.016	0.57	0.19
	at. %	0.13	0.49	1.33	0.39	0.018	0.97	0.028	0.33	0.20
Plate B	wt %	0.17	0.64	1.39	0.21	0.013	0.23	0.013	0.50	—
	at. %	0.15	0.60	1.40	0.41	0.023	1.06	0.022	0.29	—
Forging	wt %	0.02	0.76	0.72	0.21	0.014	0.24	0.012	0.62	0.34
	at. %	0.017	0.72	0.72	0.41	0.025	1.11	0.021	0.36	0.36
Weld A	wt %	0.28	0.59	1.49	0.51	0.016	0.09	0.016	0.39	0.06
	at. %	0.24	0.56	1.5	1.01	0.03	0.42	0.03	0.23	0.06
Weld B	wt %	0.30	0.58	1.63	0.61	0.017	0.08	0.012	0.39	0.10
	at. %	0.26	0.55	1.64	1.20	0.03	0.37	0.021	0.22	0.10

Table 2. Thermal history of as-received (reference) and long-term thermally aged commercial alloys

Material type	Heat number	Heat treatment <sup>a</sup> (as-received)	Thermal aging condition
Plate A	C5114-1	Austenitized 899–927°C for 1 h/in., WQ Tempered 649°C for 1 h/in., AC Stress-relieved 593–621°C for 29 h, FC	93,000 h Arkansas Unit-1 280°C
Plate B HSST 02	A1195-1	Austenitized 829–913°C for 4 h, WQ Tempered 649–677°C for 4 h, FC Stress-relieved 593–621°C for 40 h, FC	93,000 h, Arkansas Unit-1 280°C
Forging	ANK-191	Austenitized 854–877°C for 4 h, WQ Tempered 666–688°C for 10 h, WQ Stress-relieved 593–621°C for 30 h, FC	103,000 h, Oconee Unit-3 282°C
Weld A	WF-193	Stress-relieved 593–621°C for 29 h, FC	93,000 h Arkansas Unit-1 280°C
Weld B	WF-209-1	Stress-relieved 593–621°C for 30 h, FC	103,000 h Oconee Unit-3 282°C

<sup>a</sup>(WQ = water quench, AC = air cool, FC = furnace cool).

scale.<sup>18,19</sup> The microstructural characterizations were performed in the ORNL energy-compensated IPFIM.<sup>20</sup>

The experimental conditions required to obtain accurate APFIM data are well known for these ferritic steels. In particular, it is necessary to cool down the specimen to a temperature of 50 K to avoid a systematic error on the copper-level measurement. Field ion specimens were electropolished using standard procedures<sup>18</sup> from blanks that were cut from Charpy specimens. All compositions reported in this work are quoted in atomic percent.

## Results

A parallel set of experiments has been undertaken with unaged and thermally aged materials. The results concerning the chemical compositions of the ferritic matrix are summarized in Table 3. Concentration uncertainties ( $2\sigma$ ) result from counting statistics, as given by the standard deviation  $\sigma = [X(1 - X)/N]^{1/2}$ , where  $X$  is the measured concentration of an element and  $N$  is the number of atoms collected in each analysis.<sup>19</sup> It must be noted that these reactor pressure vessel steels can exhibit significant compositional variation from one specimen to another, particularly because only a small volume of material ( $\approx 3 \times 10^{-25} \text{ m}^3$ ) is sampled from any given specimen. The values in Table 3 are an average of several experiments in which  $N$  is typically  $\sim 60,000$  atoms.

Because copper repartitioning is a major contributing factor in the embrittlement of pressure vessel steels, particular attention has been paid to this element. The composition variation was particularly evident in the measurement of the copper level in the plates A and B. Severe fluctuations in the copper content from one specimen to another were observed, varying from 0.02 to 0.14 at. % Cu. However, the average copper solute concentration determined in the ferritic matrix is consistent with the nominal level for the two plates and the forging materials. On the other hand, a depletion of copper was observed in the matrix of the weld A and weld B metals for both unaged and thermally aged samples. This depletion cannot be due to the spatial fluctuation mentioned above because only 70% of the nominal level was detected.

A coarser microstructural characterization with the techniques of optical metallography and analytical transmission electron microscopy has been performed on the weld A material.<sup>21</sup> Results from the literature show that the weld metal contains mixed equiaxed and dendritic grains. The steel contains predominantly mixtures of acicular ferrite and ferrite-carbide aggregate totaling about 97% of the microstructure of the material. This alloy shows the presence of large, randomly distributed, spherical inclusions containing primarily Mn and Si. The average measured composition of these Mn-containing precipitates indicates the presence of 0.2 at. % Cu in the precipitates. The microstructure also exhibits rounded elongated precipitates, identified as  $M_2C$  carbides, which are located primarily on grain boundaries with only occasional carbides within the grains. The average composition of these carbides indicates the presence of 0.6 at. % Cu in the precipitates. Small  $M_2C$ -type carbides were also found, again mostly on grain boundaries, randomly distributed, but generally associated with other precipitates. The average composition indicates the presence of 0.4 at. % Cu in these carbides. However, the low level of copper encountered in these different features cannot fully account for the measured copper depletion from the nominal  $\sim 0.25$  to  $\sim 0.17$  at. % Cu measured in the matrix.

The stress relief heat treatment for the weld A and weld B materials was performed at a temperature between 593 and 621 °C. The predicted solubility limit of copper in the iron-copper binary system ranges between 0.17 and 0.24 at. % for these two temperatures. The detected value of 0.17 at. % can be explained by the solubility limit of copper in the ferritic solid solution at the stress-relief treatment. This suggests the formation of other copper-rich precipitates during the stress-relief heat treatment, or

Table 3. Chemical compositions (at. %) of the ferritic matrix determined by APFIM in unaged (UnA) and long-term thermally aged (Th-A) materials<sup>a</sup>

	Plate A		Plate B		Forging		Weld A		Weld B	
	UnA	Th-A	UnA	Th-A	UnA	Th-A	UnA	Th-A	UnA	Th-A
Cu	0.11 ± 0.05	0.09 ± 0.03	0.09 ± 0.05	0.07 ± 0.03	0.03 ± 0.01	0.03 ± 0.02	0.14 ± 0.03	0.15 ± 0.05	0.16 ± 0.03	0.17 ± 0.02
Ni	0.64 ± 0.13	0.74 ± 0.10	0.74 ± 0.15	0.50 ± 0.11	0.72 ± 0.07	0.63 ± 0.10	0.45 ± 0.06	0.45 ± 0.09	0.42 ± 0.05	0.43 ± 0.05
Mn	0.95 ± 0.15	1.30 ± 0.13	0.64 ± 0.14	1.05 ± 0.17	0.48 ± 0.06	0.53 ± 0.09	1.20 ± 0.10	0.90 ± 0.12	1.24 ± 0.09	1.22 ± 0.09
Si	0.41 ± 0.10	0.62 ± 0.09	0.44 ± 0.12	0.43 ± 0.10	0.50 ± 0.06	0.44 ± 0.08	1.05 ± 0.10	0.80 ± 0.12	1.73 ± 0.10	1.49 ± 0.09
P	0.02 ± 0.02	0.003 ± 0.003	0.01 ± 0.01	0.007 ± 0.007	0.02 ± 0.01	0.003 ± 0.003	0.03 ± 0.03	0.02 ± 0.02	0.05 ± 0.02	0.07 ± 0.02
C	— —	0.006 ± 0.006	0.02 ± 0.02	— —	0.004 ± 0.004	— —	0.005 ± 0.005	— —	0.008 ± 0.008	0.03 ± 0.01
Mo	0.11 ± 0.05	0.17 ± 0.04	0.12 ± 0.06	0.15 ± 0.06	0.12 ± 0.03	0.08 ± 0.03	0.18 ± 0.04	0.14 ± 0.05	0.20 ± 0.04	0.23 ± 0.04
Cr	0.11 ± 0.05	0.06 ± 0.02	0.04 ± 0.04	0.08 ± 0.05	0.15 ± 0.03	0.20 ± 0.05	0.03 ± 0.03	0.03 ± 0.01	0.07 ± 0.02	0.06 ± 0.02

<sup>a</sup>All compositions are the average of several experiments ( $\pm 2\sigma$ ).

also during the furnace cooling period (at a cooling rate of  $\sim 8^\circ\text{C}/\text{h}$  to  $\sim 310^\circ\text{C}$ ), even though they have not been detected in this investigation. However, coarse copper-enriched precipitates have been detected at grain boundaries of A533B steels by APFIM.<sup>7</sup>

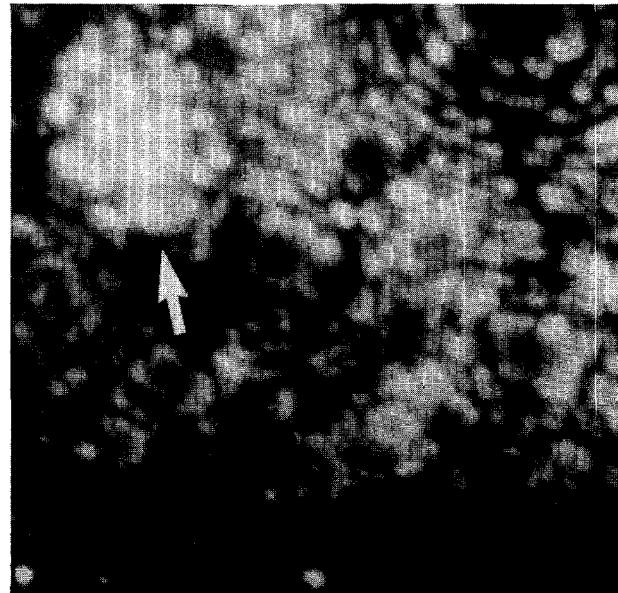
The Si and Ni contents of the matrix are, within the standard deviation, in good agreement with the nominal composition of the alloy. The depletion of carbon and molybdenum in the materials suggests the presence of a high volume fraction of carbides, particularly in plates A and B and the forging materials. This high volume fraction of carbides in these materials may also explain the observed phosphorus matrix depletion. Indeed, phosphorus is often encountered at ferrite-carbide interfaces. In all materials, the detected level of manganese is always slightly lower than the nominal concentration. This depletion is due to its presence in cementite carbides, as shown below.

Analyses were performed on both the carbides and the matrix to characterize the structure of these unaged and long-term thermally aged materials. The more common features encountered in the atom probe analyses of these steels are  $\text{M}_3\text{C}$  cementite carbides and molybdenum-containing carbides.

Molybdenum-containing carbides were frequently observed in both unaged and thermally aged specimens. The small carbides have a disc, needle, or spherical shape, whereas the larger precipitates are generally spherical, as shown in Figures 1 and 2. The sizes of the observed carbides were determined to be between 5 to 20 nm. The core composition of these particles is given in Table 4. The large uncertainties for these features are due to the small numbers of ions collected from such small precipitates. The Mo:C ratio, in both the unaged and thermally aged materials, is close to that of  $\text{Mo}_2\text{C}$ .



**Figure 1. Field ion micrograph of disk-shaped molybdenum carbides in the weld B surveillance material.**



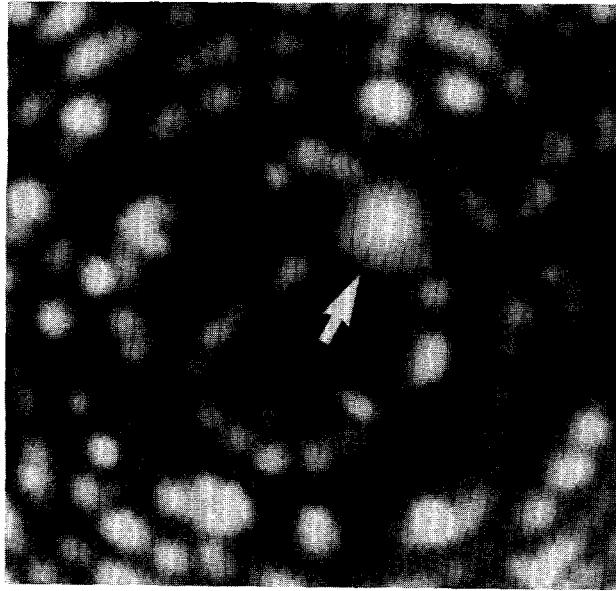
**Figure 2. Field ion micrograph of spherical molybdenum carbides in the weld B surveillance material.**

**Table 4. Composition of intragranular molybdenum-rich carbides in unaged or long-term thermally aged materials**

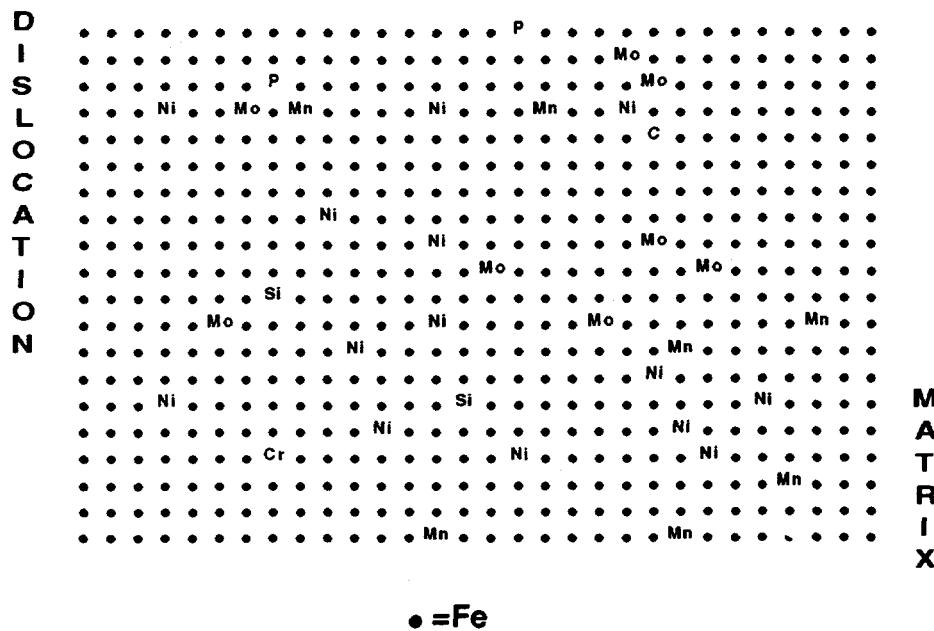
Element	% at	$2\sigma$
Mo	63.6	16.7
Cr	3.0	3.0
C	33.4	16.4

atoms during the atom probe analysis of the core of the dislocation is shown in Figure 4. Each line represents the number of atoms detected per two atomic planes of materials field evaporated from the specimen. This figure clearly indicates that Mo, P, and C are detected in the vicinity of the dislocation. The concentrations of these elements decrease significantly in regions far removed from the dislocation.

In addition, molybdenum atoms were also detected in the vicinity of dislocations, sometimes associated with carbon and phosphorus, as evident in Figures 3 and 4. A field ion image of a dislocation having a Burgers vector component normal to the specimen surface is shown in Figure 3. The presence of a dislocation converts the usual pattern of concentric rings at a crystallographic pole into a spiral at its point of emergence on the specimen surface.<sup>22</sup> Bright spots, characteristic of molybdenum atoms, that decorate the dislocation can be observed near its point of emergence. The sequence of evaporation of the



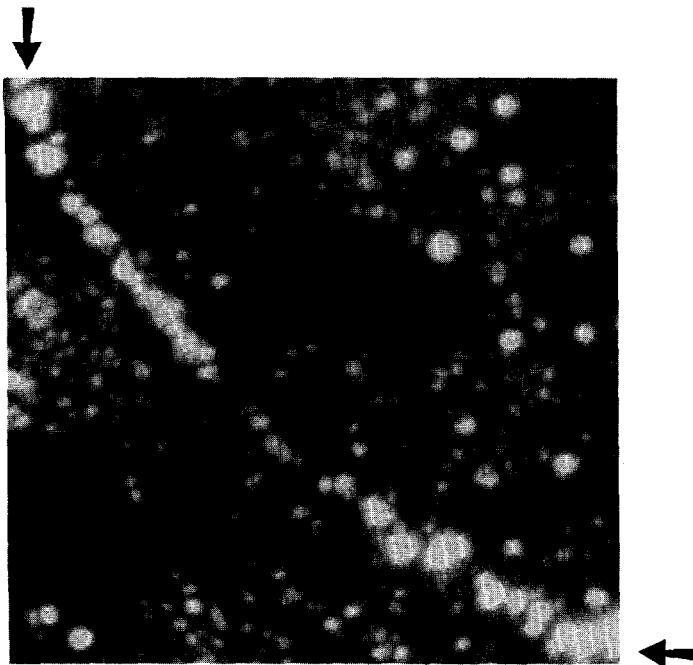
**Figure 3. Field ion micrograph of a decorated dislocation in the unaged forging material. Bright and diffuse spots are molybdenum or phosphorus.**



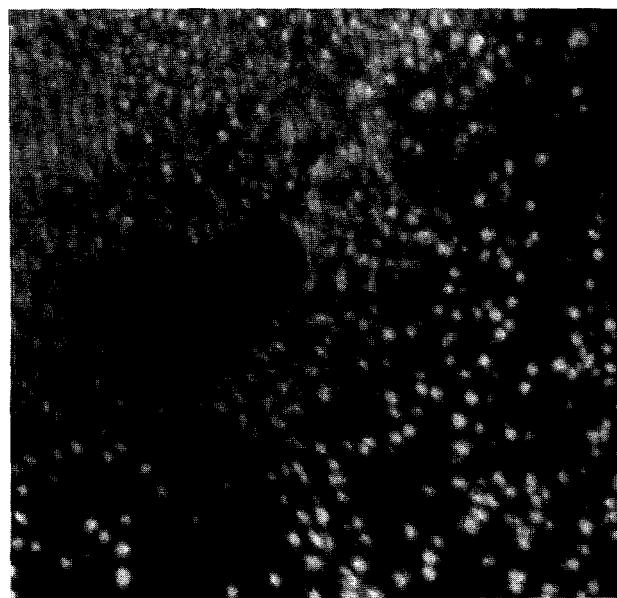
**Figure 4. Sequence of arrival of ions at the detector from the analyzed dislocation (see Figure 3). Note the Mo, C, and P enrichments in the core of the dislocation. Each line represents the removal of approximately two atomic planes of material from the specimen.**

In addition to the  $\text{Mo}_2\text{C}$  carbides and the cores of decorated dislocations, molybdenum is also observed at the grain boundaries. A field ion image of a grain boundary observed in the long-term thermally aged weld B material is shown in Figure 5. This micrograph clearly indicates that the grain boundary is decorated with a 1- to 2-nm-thick layer of molybdenum carbide precipitates. This type of decoration has been observed in various Russian and Western steels and is a common feature of molybdenum-containing pressure vessel steels.<sup>7,8,23</sup>

Another common feature encountered in these materials is the presence of  $\text{M}_3\text{C}$  cementite carbides. A field ion micrograph of a cementite-ferrite interface observed in the thermally aged Plate B material is shown in Figure 6. The ferrite can be easily recognized by the presence of crystallographic poles (i.e., families of concentric rings), which are not evident in the darkly imaging cementite phase. A brightly imaging molybdenum carbide precipitate is also evident at the interface. A composition profile starting in this molybdenum carbide and emerging immediately into a cementite precipitate located at the interface is shown in Figure 7. It is evident from this composition profile that there is a large Fe and Mn content in the cementite but little solubility of the Fe, Cr, or Mn alloying elements in the  $\text{Mo}_2\text{C}$  precipitate. Analyses were performed in unaged and thermally aged specimens to determine whether there was an effect of the ~10 years thermal-aging treatment on the evolution of the composition of cementite. Analyses were successful in the Plate A for the unaged specimen and in the Plate B for the thermally aged one. The chemical compositions and stress-relief heat treatment of these two materials are so similar that they can be compared. In addition, the atom probe results were compared with



**Figure 5. Decorated grain boundary in the weld B long-term thermally aged surveillance material. Bright spots are ultrafine  $\text{Mo}_2\text{C}$  carbides.**

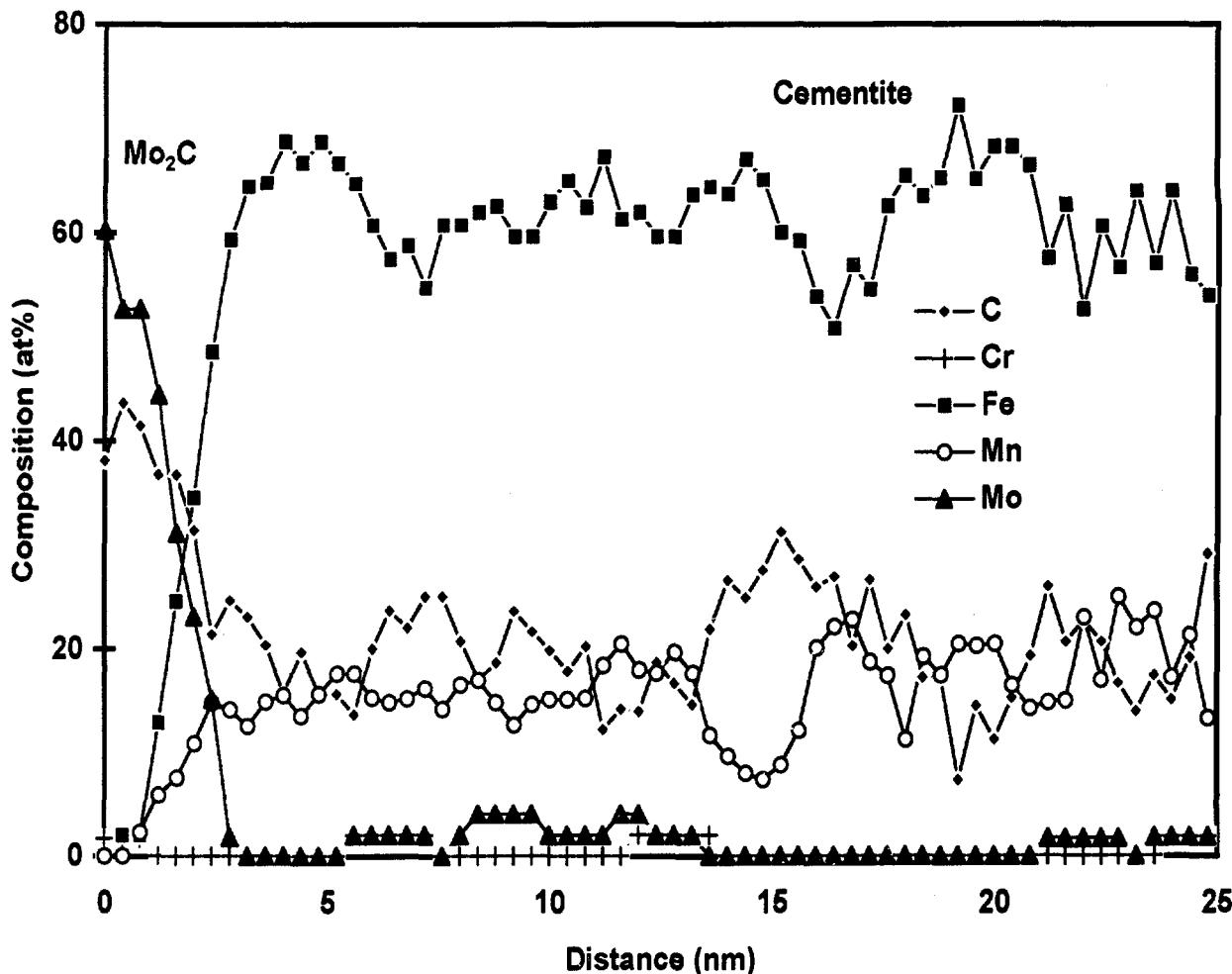


**Figure 6. Darkly imaging cementite and brightly imaging ferrite in long-term thermally aged plate B material. Bright region at the interface is  $\text{Mo}_2\text{C}$  carbide (see Figure 7).**

thermodynamic predictions.<sup>24</sup> Thermocalc™ calculations were performed at two temperatures (620 and 593°C) within the stress-relief heat treatment and also at 300°C, the temperature at which specimens were thermally aged (93,000 h in the case of the Plate B). The results are summarized in Table 5.

The atom probe results revealed, in both unaged and thermally aged materials, carbides with classic  $\text{M}_3\text{C}$  stoichiometry, where M stands for Fe and Mn (in majority), Mo, Cr, and Ni (in minority) and also Cu and V (as traces, 0.02 at. %). These results are similar to those observed in previous analyses of the Chooz A pressure vessel steel<sup>9</sup> and weld metal.<sup>21</sup> In addition, the experimental compositions are in good agreement with the Thermocalc™ predictions for materials aged at 593 to 620°C. In all cases, the compositions are comparable to the predicted values at ~600°C. This agreement indicates that long-term thermal aging has no significant impact on the evolution of the microchemistry of cementite carbides.

These initial APFIM microstructural examinations performed on unaged and long-term, low-temperature, thermally aged materials show no significant evolution of the structure of the material. No phase transformation has been observed for this low-temperature heat treatment. Unaged materials and materials thermally aged for approximately 10 years have a similar ferritic matrix chemistry and carbide compositions. These results are consistent with the observed mechanical properties of these materials.<sup>17</sup> A general review of the data on thermally aged material indicates virtually no significant change in the impact data. Only small variations in the Charpy V-notch impact properties were observed for all materials after exposure at the thermal-aging temperature. Small



**Figure 7. Composition profile through a molybdenum carbide and cementite in long-term thermally aged plate B surveillance material.**

increases ( $\sim 1^\circ\text{C}$ ) in transition temperature were observed for the forging metal and weld B surveillance materials aged at 103,000 h, and their upper-shelf energies demonstrated small decreases (3 to 11 J). The Charpy V-notch results for plate A and weld metal A aged for 93,000 h revealed differences in that the 41-J transition temperatures for both materials decreased slightly.

Also, and especially for the weld materials, the copper remains in solid solution with a concentration following the solubility of copper in iron for unaged specimens. A metastable solid solution is observed for thermally aged specimens. This confirms that the thermal mobility of copper in iron at  $300^\circ\text{C}$  is effectively zero.

## Conclusion

Microstructural characterization of long-term ( $\sim 100,000$  h) thermally aged ( $300^\circ\text{C}$ ) and unaged surveillance materials obtained from the B&W Owners Group was performed. Two welds, two plates, and one forging material were investigated. The comparison between the thermally aged materials and unaged materials permitted, for the first time, the investigation of a potential thermal-aging effect. Although a general review of the thermal-aging data indicates that there may be some propensity toward embrittlement in sensitive materials,<sup>11,12</sup> the materials examined in this study did not exhibit any

Table 5. Atom probe analyses of the cementite carbides. Comparison of the atom probe results with the Thermocalc™ predictions

	Fe	C	Mn	Mo	Cr	Ni
<b>Plate A Unaged</b> Stress-relieved 593–621°C for 29 h, FC						
Thermocalc prediction (620°C)	59.1	25.0	11.1	1.5	3.1	0.1
Thermocalc prediction (593°C)	57.5	25.0	12.5	1.6	3.3	0.1
Atom probe experiment (593–620°C)	64 ± 0.8	25.6 ± 0.7	8.7 ± 0.5	1.2 ± 0.2	0.5 ± 0.1	– –
Thermocalc prediction (300°C)	36.5	25.0	29.9	3.4	5.0	0.1
<b>Plate B Long-term thermally aged</b> Stress-relieved 593–621°C for 40 h, FC 93,000 h at 280°C						
Thermocalc prediction (620°C)	61.8	25.0	11.6	1.5	–	0.1
Thermocalc prediction (593°C)	60.4	25.0	12.9	1.5	–	0.1
Thermocalc prediction (300°C)	42.1	25.0	29.3	3.35	–	0.2
Atom probe experiment 93,000 h @ 280°C	61.1 ± 0.1	25.4 ± 0.9	11.9 ± 0.7	0.9 ± 0.2	–	0.2 ± 0.1

significant embrittlement or microstructural evolution. The same matrix copper level was found before and after the long thermal-aging treatment. In the two welds, a significant decrease of the copper level in the matrix over the nominal bulk composition was found and is due to copper precipitation during the stress-relief heat treatment. This APFIM comparison of the microstructures in all three conditions is consistent with the measured mechanical properties (transition temperature shift); i.e., no significant changes in either the microstructure or the mechanical properties has been observed.

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## BIBLIOGRAPHIC DATA SHEET

(See instructions on the reverse)

1. REPORT NUMBER  
(Assigned by NRC, Add Vol., Supp., Rev., and Addendum Numbers, if any.)NUREG/CR-6537  
ORNL/TM-13406

## 2. TITLE AND SUBTITLE

Influence of Long-Term Thermal Aging on the Microstructural Evolution of Nuclear Reactor Pressure Vessel Materials

An Atom Probe Study

## 3. DATE REPORT PUBLISHED

MONTH                   YEAR  
March                   1998

## 4. FIN OR GRANT NUMBER

L1098

## 5. AUTHOR(S)

P. Pareige\*, K. F. Russell, R. E. Stoller, M. K. Miller

## 6. TYPE OF REPORT

Technical

## 7. PERIOD COVERED (Inclusive Dates)

## 8. PERFORMING ORGANIZATION - NAME AND ADDRESS (If NRC, provide Division, Office or Region, U.S. Nuclear Regulatory Commission, and mailing address; if contractor, provide name and mailing address.)

Oak Ridge National Laboratory  
Oak Ridge, TN 37831-6285\*Laboratoire de Microscopie Ionique et Electronique, GMP-UMR 6634  
Faculte des Sciences de l'Universite de Rouen  
76128 Mont Saint AIGNAN, Cedex, France

## 9. SPONSORING ORGANIZATION - NAME AND ADDRESS (If NRC, type "Same as above"; if contractor, provide NRC Division, Office or Region, U.S. Nuclear Regulatory Commission, and mailing address.)

Division of Engineering Technology  
Office of Nuclear Regulatory Research  
U.S. Nuclear Regulatory Commission  
Washington, DC 20555-0001

## 10. SUPPLEMENTARY NOTES

M. G. Vassilaros, NRC Project Manager

## 11. ABSTRACT (200 words or less)

Atom probe field ion microscopy (APFIM) investigations of the microstructure of unaged (as-fabricated) and long-term thermally aged (~100,000 h at 280°C) surveillance materials from commercial reactor pressure vessel steels were performed. This combination of materials and conditions permitted the investigation of potential thermal-aging effects. This microstructural study focused on the quantification of the compositions of the matrix and carbides. The APFIM results indicate that there was no significant microstructural evolution after a long-term thermal exposure in weld, plate, or forging materials. The matrix depletion of copper that was observed in weld materials was consistent with the copper concentration in the matrix after the stress-relief heat treatment. The compositions of cementite carbides aged for 100,000 h were compared with the Thermocalc™ prediction. The APFIM comparisons of materials under these conditions are consistent with the measured change in mechanical properties such as the Charpy transition temperature.

## 12. KEY WORDS/DESCRIPTORS (List words or phrases that will assist researchers in locating the report.)

pressure vessels  
atom probe  
thermal aging  
microstructural  
embrittlement  
cementite carbide  
copper

## 13. AVAILABILITY STATEMENT

unlimited

## 14. SECURITY CLASSIFICATION

(This Page)

unclassified

(This Report)

unclassified

## 15. NUMBER OF PAGES

## 16. PRICE