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# Long-Term Embrittlement of Cast Duplex Stainless Steels in LWR Systems

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Prepared by O. K. Chopra, H. M. Chung

Argonne National Laboratory

Prepared for  
U.S. Nuclear Regulatory Commission

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*Long-Term Embrittlement of Cast Duplex Stainless Steels in LWR Systems: Annual Report October 1983-September 1984*, NUREG/CR-4204, ANL-85-20 (March 1985).

*Long-Term Embrittlement of Cast Duplex Stainless Steels in LWR Systems: Annual Report October 1984-September 1985*, NUREG/CR-4503, ANL-86-3 (January 1986).

*Long-Term Embrittlement of Cast Duplex Stainless Steels in LWR Systems: Semiannual Report October 1985-March 1986*, NUREG/CR-4744 Vol. I, No. 1, ANL-86-54 (September 1986).

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# **Long-Term Embrittlement of Cast Duplex Stainless Steels in LWR Systems**

by

O. K. Chopra and H. M. Chung

## **Abstract**

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This progress report summarizes work performed by Argonne National Laboratory on long-term embrittlement of cast duplex stainless steels in LWR systems during the six months from October 1988 to March 1989. Charpy-impact data are presented for several heats of cast stainless steel aged at temperatures between 320 and 450°C for times up to 30,000 h. Thermal aging decreases impact energy and shifts transition curves to higher temperatures. A saturation effect is observed for room-temperature impact energy and upper-shelf energy. Charpy data are analyzed to obtain the activation energy of the kinetics of embrittlement. The results suggest that the activation energy of embrittlement is not constant in the temperature range of 290–400°C, but increases as temperature decreases. A correlation is presented for estimating the extent of embrittlement of cast stainless steels from known material parameters. The degradation in mechanical properties can be reversed by annealing the embrittled material for 1 h at 550°C and then water quenching.



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## Executive Summary

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Cast stainless steels used in pump casings, valve bodies, piping, and other components in coolant systems of light-water nuclear reactors (LWRs) suffer loss of toughness after many years of service at 290–320°C. A program is being conducted to investigate the low-temperature embrittlement of cast duplex stainless steels under LWR operating conditions and to evaluate possible remedies for embrittlement problems in existing and future plants. The scope of the investigation includes the following goals: (1) characterize and correlate the microstructure of in-service reactor components and laboratory-aged material with loss of fracture toughness to establish the mechanism of aging and validate the simulation of in-reactor degradation by accelerated aging, (2) establish the effects of key compositional and metallurgical variables on the kinetics and extent of embrittlement, and (3) develop the methodology and correlations for predicting the toughness loss suffered by cast stainless steel components during normal and extended life of LWRs.

Microstructural and mechanical-property data are being obtained on 25 experimental heats (19 in the form of static-cast keel blocks and 6 in the form of slabs) and 6 commercial heats (centrifugally cast pipes and a static-cast pump impeller and pump casing ring), as well as on reactor-aged material of grades CF-3, CF-8, and CF-8M cast stainless steel. The ferrite content of the cast materials ranges from 3 to 30%. Ferrite morphology for the castings containing >5% ferrite is either lacy or acicular.

Charpy-impact, tensile, and J-R curve tests have been conducted on several experimental and commercial heats of cast stainless steel that were aged up to 30,000 h at 290–400°C. Results indicate that thermal aging at these temperatures increases the tensile strength and decreases the impact energy and fracture toughness of the steels. The Charpy transition curve shifts to higher temperatures. Different heats exhibit different degrees of embrittlement. In general, the low-carbon CF-3 steels are the most resistant, and the molybdenum-containing high-carbon CF-8M steels are the least resistant to embrittlement. Embrittlement of cast stainless steels results in brittle fracture associated with either cleavage of the ferrite or separation of the ferrite/austenite phase boundary. Predominantly brittle failure occurs when either the ferrite phase is continuous, e.g., in cast material with a large ferrite content, or the ferrite/austenite phase boundary provides an easy path for crack propagation, e.g., in high-C grades of cast steel with large phase-boundary carbides. Consequently, the amount, size, and distribution of the ferrite phase in the duplex structure and the presence of phase-boundary carbides are important parameters in controlling the degree or extent of embrittlement.

Thermal aging of cast stainless steels at 300–450°C leads to precipitation of additional phases in the ferrite matrix, e.g., formation of a Cr-rich  $\alpha'$  phase by spinodal decomposition; nucleation and growth of  $\alpha'$ ; precipitation of Ni- and Si-rich G phase,  $M_{23}C_6$ , and  $\gamma_2$  (austenite); and additional precipitation and/or growth of existing carbides at the ferrite/austenite phase boundaries. The additional phases provide the strengthening mechanisms that increase strain hardening and local tensile stress. Consequently, the critical stress level for brittle fracture is achieved at higher temperatures. The effects of material variables on the embrittlement of cast stainless steels have been evaluated.

This report presents Charpy-impact data on several experimental and commercial heats of cast stainless steel that were aged up to 30,000 h at 290–450°C. The results indicate that, for a specific cast stainless steel, the extent of embrittlement, i.e., the minimum room-temperature impact energy that would be achieved after long-term aging, depends on the chemical composition and ferrite morphology of the steel. A correlation is presented for estimating the extent of embrittlement from known material information. The extent and rate of embrittlement depend on material parameters, i.e., ferrite morphology and chemical composition of the steel. Ferrite morphology strongly affects the extent of embrittlement, whereas material composition influences the kinetics of embrittlement. Small changes in the constituent elements of the cast material can cause the kinetics of embrittlement to vary significantly.

Annealing studies indicate that the degradation in mechanical properties due to thermal embrittlement can be reversed by annealing the embrittled material for 1 h at 550°C and then water quenching. The kinetics of embrittlement can also be obtained from aging studies on the reembrittlement of recovery-annealed material. This procedure may be useful for establishing baseline mechanical properties and kinetics of embrittlement of service-aged components.

# 1 Introduction

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A program is being conducted to investigate the significance of low-temperature embrittlement of cast duplex stainless steels under light-water reactor (LWR) operating conditions and to evaluate possible remedies for embrittlement problems in existing and future plants. The scope of the program includes the following goals: (1) characterize and correlate the microstructure of in-service reactor components and laboratory-aged material with loss of fracture toughness to establish the mechanism of aging and validate the simulation of in-reactor degradation by accelerated aging, (2) establish the effects of key compositional and metallurgical variables on the kinetics and extent of embrittlement, and (3) develop the methodology and correlations for predicting the toughness loss suffered by cast stainless steel components during normal and extended life of LWRs.

Microstructural and mechanical-property data are being obtained on 25 experimental heats (19 in the form of static-cast keel blocks and 6 in the form of 76-mm slabs) and 6 commercial heats (centrifugally cast pipes and a static-cast pump impeller and pump casting ring) as well as reactor-aged material of grades CF-3, CF-8, and CF-8M cast stainless steel. Specimen blanks for Charpy-impact, tensile, and J-R curve tests are being aged at 290, 320, 350, 400, and 450°C for times up to 50,000 h. The reactor-aged material is from the recirculating-pump cover plate assembly of the KRB reactor, which was in service in Gundremmingen, West Germany, for  $\approx$ 12 yr ( $\approx$ 8 yr at a service temperature of 284°C). Fractured impact test bars from five heats of aged cast stainless steel were obtained from the Georg Fischer Co. (GF) of Switzerland for microstructural characterization. The materials from GF are from a previous study of long-term aging behavior of cast stainless steel.<sup>1</sup> The data on chemical composition, ferrite content, hardness, ferrite morphology, and grain structure of the experimental and commercial heats have been reported earlier.<sup>2-6</sup> The chemical composition, hardness, and ferrite content and distribution of some of the cast materials are given in Table 1. The results of microstructural characterization and mechanical-property data from Charpy-impact, tensile, and J-R curve tests on 16 heats of cast stainless steel aged up to 30,000 h at temperatures between 290 and 450°C have also been presented earlier.<sup>7-15</sup> A preliminary assessment of the processes and significance of thermal aging in cast stainless steels were also presented.<sup>10-12</sup>

The results indicate that thermal aging at temperatures below 500°C increases the tensile strength and decreases the impact energy and fracture toughness of the steels. The Charpy transition curve shifts to higher temperatures. Different heats exhibit different degrees of embrittlement. For cast stainless steel of all grades, the extent of embrittlement increases with an increase in ferrite content. The low-C CF-3 steels are the most resistant, and the Mo-containing, high-C CF-8M steels are the least resistant to embrittlement.

The mechanisms of embrittlement of cast duplex stainless steel have been discussed.<sup>12</sup> Embrittlement of cast stainless steel results in brittle fracture associated with either cleavage of the ferrite or separation of the ferrite/austenite phase boundary (Fig. 1). The degree of embrittlement is controlled by the amount of brittle fracture. Cast stainless steels with poor impact strength exhibit >80% brittle fracture. In some cast steels, a fraction of the material may fail in a brittle fashion but the surrounding austenite provides ductility and toughness. Such steels have adequate impact strength even after long-term aging.

Table 1. Product form, chemical composition, hardness, and ferrite morphology of various heats of cast stainless steel

| Heat                                  | Grade | Chemical Composition (wt.%) |      |       |       |      |       |       | Ferrite <sup>a</sup><br>(%) |       | Hard-<br>ness<br>RB | Ferrite<br>Spacing<br>(μm) |      |     |
|---------------------------------------|-------|-----------------------------|------|-------|-------|------|-------|-------|-----------------------------|-------|---------------------|----------------------------|------|-----|
|                                       |       | Mn                          | Si   | P     | S     | Mo   | Cr    | Ni    | N                           | C     | Calc.               | Meas.                      |      |     |
| <u>Keel Blocks<sup>b</sup></u>        |       |                             |      |       |       |      |       |       |                             |       |                     |                            |      |     |
| 50                                    | CF-3  | 0.60                        | 1.10 | 0.016 | 0.007 | 0.33 | 17.89 | 9.14  | 0.079                       | 0.034 | 3.0                 | 4.4                        | 80.1 | 194 |
| 49                                    | CF-3  | 0.60                        | 0.95 | 0.010 | 0.007 | 0.32 | 19.41 | 10.69 | 0.065                       | 0.010 | 4.4                 | 7.2                        | 76.6 | 185 |
| 48                                    | CF-3  | 0.60                        | 1.08 | 0.009 | 0.006 | 0.30 | 19.55 | 10.46 | 0.072                       | 0.011 | 5.1                 | 8.7                        | 78.1 | 127 |
| 47                                    | CF-3  | 0.60                        | 1.06 | 0.007 | 0.006 | 0.59 | 19.81 | 10.63 | 0.028                       | 0.018 | 8.4                 | 16.3                       | 79.7 | 68  |
| 52                                    | CF-3  | 0.57                        | 0.92 | 0.012 | 0.005 | 0.35 | 19.49 | 9.40  | 0.052                       | 0.009 | 10.3                | 13.5                       | 81.6 | 69  |
| 51                                    | CF-3  | 0.63                        | 0.86 | 0.014 | 0.005 | 0.32 | 20.13 | 9.06  | 0.058                       | 0.010 | 14.3                | 18.0                       | 83.8 | 52  |
| 58                                    | CF-8  | 0.62                        | 1.12 | 0.010 | 0.005 | 0.33 | 19.53 | 10.89 | 0.040                       | 0.056 | 3.2                 | 2.9                        | 77.1 | 303 |
| 54                                    | CF-8  | 0.55                        | 1.03 | 0.011 | 0.005 | 0.35 | 19.31 | 9.17  | 0.084                       | 0.063 | 4.1                 | 1.8                        | 83.3 | 317 |
| 57                                    | CF-8  | 0.62                        | 1.08 | 0.009 | 0.004 | 0.34 | 18.68 | 9.27  | 0.047                       | 0.056 | 4.4                 | 4.0                        | 80.2 | 138 |
| 53                                    | CF-8  | 0.64                        | 1.16 | 0.012 | 0.009 | 0.39 | 19.53 | 9.23  | 0.049                       | 0.065 | 6.3                 | 8.7                        | 83.1 | 92  |
| 56                                    | CF-8  | 0.57                        | 1.05 | 0.007 | 0.007 | 0.34 | 19.65 | 9.28  | 0.030                       | 0.066 | 7.3                 | 10.1                       | 82.5 | 84  |
| 59                                    | CF-8  | 0.60                        | 1.08 | 0.008 | 0.007 | 0.32 | 20.33 | 9.34  | 0.045                       | 0.062 | 8.8                 | 3.5                        | 83.2 | 75  |
| 61                                    | CF-8  | 0.65                        | 1.01 | 0.007 | 0.007 | 0.32 | 20.65 | 8.86  | 0.080                       | 0.054 | 10.0                | 13.1                       | 85.3 | 82  |
| 60                                    | CF-8  | 0.67                        | 0.95 | 0.008 | 0.006 | 0.31 | 21.05 | 8.34  | 0.058                       | 0.064 | 15.4                | 21.1                       | 86.7 | 63  |
| 62                                    | CF-8M | 0.72                        | 0.56 | 0.007 | 0.005 | 2.57 | 18.29 | 12.39 | 0.030                       | 0.063 | 2.8                 | 4.5                        | 78.1 | 140 |
| 63                                    | CF-8M | 0.61                        | 0.58 | 0.007 | 0.006 | 2.57 | 19.37 | 11.85 | 0.031                       | 0.055 | 6.4                 | 10.4                       | 81.6 | 81  |
| 66                                    | CF-8M | 0.60                        | 0.49 | 0.012 | 0.007 | 2.39 | 19.45 | 9.28  | 0.029                       | 0.047 | 19.6                | 19.8                       | 85.3 | 41  |
| 65                                    | CF-8M | 0.50                        | 0.48 | 0.012 | 0.007 | 2.57 | 20.78 | 9.63  | 0.064                       | 0.049 | 20.9                | 23.4                       | 89.0 | 43  |
| 64                                    | CF-8M | 0.60                        | 0.63 | 0.006 | 0.005 | 2.46 | 20.76 | 9.40  | 0.038                       | 0.038 | 29.0                | 28.4                       | 89.7 | 41  |
| <u>76-mm Slabs<sup>c</sup></u>        |       |                             |      |       |       |      |       |       |                             |       |                     |                            |      |     |
| 69                                    | CF-3  | 0.63                        | 1.13 | 0.015 | 0.005 | 0.34 | 20.18 | 8.59  | 0.028                       | 0.023 | 21.0                | 23.6                       | 83.7 | 35  |
| 73                                    | CF-8  | 0.72                        | 1.09 | 0.028 | 0.016 | 0.25 | 19.43 | 8.54  | 0.053                       | 0.070 | 7.0                 | 7.7                        | 78.8 | 253 |
| 68                                    | CF-8  | 0.64                        | 1.07 | 0.021 | 0.014 | 0.31 | 20.64 | 8.08  | 0.062                       | 0.063 | 14.9                | 23.4                       | 84.6 | 87  |
| 70                                    | CF-8M | 0.55                        | 0.72 | 0.021 | 0.016 | 2.30 | 19.17 | 9.01  | 0.049                       | 0.066 | 14.2                | 18.9                       | 86.5 | 96  |
| 74                                    | CF-8M | 0.54                        | 0.73 | 0.022 | 0.016 | 2.51 | 19.11 | 9.03  | 0.048                       | 0.064 | 15.5                | 18.4                       | 85.8 | 90  |
| 75                                    | CF-8M | 0.53                        | 0.67 | 0.022 | 0.012 | 2.58 | 20.86 | 9.12  | 0.052                       | 0.065 | 24.8                | 27.8                       | 89.5 | 69  |
| <u>Reactor Components<sup>d</sup></u> |       |                             |      |       |       |      |       |       |                             |       |                     |                            |      |     |
| P3                                    | CF-3  | 1.06                        | 0.88 | 0.017 | 0.014 | 0.01 | 18.89 | 8.45  | 0.168                       | 0.021 | 2.8                 | 1.9                        | 82.2 | —   |
| P2                                    | CF-3  | 0.74                        | 0.94 | 0.019 | 0.006 | 0.16 | 20.20 | 9.38  | 0.040                       | 0.019 | 12.5                | 15.6                       | 83.8 | 69  |
| I                                     | CF-3  | 0.47                        | 0.83 | 0.030 | 0.011 | 0.45 | 20.20 | 8.70  | 0.032                       | 0.019 | 20.4                | 17.1                       | 81.0 | 65  |
| C1                                    | CF-8  | 1.22                        | 1.18 | 0.033 | 0.008 | 0.65 | 19.00 | 9.37  | 0.040                       | 0.039 | 7.8                 | 2.2                        | 79.5 | —   |
| P1                                    | CF-8  | 0.59                        | 1.12 | 0.026 | 0.013 | 0.04 | 20.49 | 8.10  | 0.056                       | 0.036 | 17.7                | 24.1                       | 84.9 | 90  |
| P4                                    | CF-8M | 1.07                        | 1.02 | 0.019 | 0.015 | 2.05 | 19.64 | 10.00 | 0.151                       | 0.040 | 5.9                 | 10.0                       | 83.1 | 182 |
| 205                                   | CF-8M | 0.93                        | 0.63 | 0.019 | —     | 3.37 | 17.88 | 8.80  | —                           | 0.040 | 21.0                | 15.9                       | —    | 79  |
| 758                                   | CF-8M | 0.91                        | 0.62 | 0.018 | —     | 3.36 | 17.91 | 8.70  | —                           | 0.030 | 24.2                | 19.2                       | —    | 62  |
| <u>Reactor-Aged<sup>e</sup></u>       |       |                             |      |       |       |      |       |       |                             |       |                     |                            |      |     |
| KRB                                   | CF-8  | 0.31                        | 1.17 | —     | —     | 0.17 | 21.99 | 8.03  | 0.038                       | 0.062 | 27.7                | 34.0                       | —    | —   |

Table 1. (Contd.)

| Heat                               | Grade | Chemical Composition (wt.%) |      |       |       |      |       |      |       | Ferrite <sup>a</sup> (%) |       | Hardness RB | Ferrite Spacing (μm) |     |
|------------------------------------|-------|-----------------------------|------|-------|-------|------|-------|------|-------|--------------------------|-------|-------------|----------------------|-----|
|                                    |       | Mn                          | Si   | P     | S     | Mo   | Cr    | Ni   | N     | C                        | Calc. | Meas.       |                      |     |
| <u>Laboratory-Aged<sup>f</sup></u> |       |                             |      |       |       |      |       |      |       |                          |       |             |                      |     |
| 280                                | CF-3  | 0.50                        | 1.37 | 0.015 | 0.006 | 0.25 | 21.60 | 8.00 | 0.038 | 0.028                    | 36.3  | 40.0        | —                    | 186 |
| 278                                | CF-8  | 0.28                        | 1.00 | 0.008 | 0.019 | 0.13 | 20.20 | 8.27 | 0.030 | 0.038                    | 18.5  | 15.0        | —                    | 174 |
| 292                                | CF-8  | 0.34                        | 1.57 | 0.018 | 0.016 | 0.13 | 21.60 | 7.52 | 0.039 | 0.090                    | 23.9  | 28.0        | —                    | —   |
| 286                                | CF-8M | 0.40                        | 1.33 | 0.044 | 0.015 | 2.44 | 20.20 | 9.13 | 0.062 | 0.072                    | 18.9  | 22.0        | —                    | 201 |

<sup>a</sup> Calculated from the composition with Hull's equivalent factor.

Measured by ferrite scope AUTO Test FE, Probe Type FSP-1.

<sup>b</sup> *Static Cast Keel Blocks*: Foundry ESCO; Size 180 x 120 x 90–30 mm.

<sup>c</sup> *Static Cast Slabs*: Foundry ESCO; Size 610 x 610 x 76 mm.

<sup>d</sup> *Centrifugally Cast Pipes*:

P3 Foundry SANDUSKY; Size 580 mm O.D., 76 mm wall.

P2 Foundry FAM, France; Size 930 mm O.D., 73 mm wall.

P1 Foundry ESCO; Size 890 mm O.D., 63 mm wall.

P4 Foundry SANDUSKY; Size 580 mm O.D., 32 mm wall.

205 Size 305 mm O.D., 25 mm wall.

*Static Cast*:

Elbow 758: Size 305 mm O.D., 30 mm wall.

Pump Impeller I: Foundry ESCO; Size 660 mm diameter.

Pump Casing C1: Foundry ESCO; Size 600 mm O.D., 57 mm wall.

<sup>e</sup> KRB Reactor Pump Cover Plate: Foundry GF; Size 890 mm diameter.

<sup>f</sup> Aged Material from George Fischer Co., Switzerland.

Predominantly brittle failure occurs when either the ferrite phase is continuous, e.g., in cast material with a large ferrite content, or the ferrite/austenite phase boundary provides an easy path for crack propagation, e.g., in high-C cast steels with large phase-boundary carbides. Consequently, the amount, size, and distribution of the ferrite phase in the duplex structure, and the presence of phase-boundary carbides are important parameters in controlling the degree or extent of embrittlement.

Thermal aging of cast stainless steels at 300–450°C leads to precipitation of additional phases in the ferrite matrix, e.g., formation of a Cr-rich  $\alpha'$  phase by spinodal decomposition; nucleation and growth of  $\alpha'$ ; precipitation of an Ni- and Si-rich G phase,  $M_{23}C_6$ , and  $\gamma_2$  (austenite); and additional precipitation and/or growth of existing carbides at the ferrite/austenite phase boundaries.<sup>13–15</sup> The additional phases provide the strengthening mechanisms that increase strain hardening and local tensile stress. Consequently, the critical stress level for brittle fracture is achieved at higher temperatures.

Phase-boundary separation generally occurs in the high-C steels because of the presence of large  $M_{23}C_6$  at the phase boundaries. For CF-8 steels, the phase-boundary carbides form during production heat treatment of the casting. Consequently, the unaged CF-8 steels exhibit low lower-shelf energy and high mid-shelf Charpy transition temperature (CTT) relative to the CF-3 steels. The fracture mode of CF-8 steels in the lower-shelf or transition-temperature regime is predominantly phase-boundary separation;<sup>7,8</sup> in contrast, the CF-3 steels show dimpled ductile failure. Fracture by phase-boundary separation is observed in only a few heats of unaged CF-8M steels and is dependent on whether the material contains phase-boundary carbides. The difference in fracture mode is reflected in yield and maximum loads obtained from the load-time traces of the instrumented Charpy tests.

The results for unaged and aged Heats 68 (CF-8) and 69 (CF-3) are shown in Fig. 2. The estimated yield and ultimate tensile stresses<sup>7,8</sup> can be determined from the axes on the right side of the figures. The results indicate the expected decrease in yield and maximum loads with increase in temperature. Thermal aging increases the loads at all temperatures. For Heat 68, the critical stress for brittle fracture is achieved before general yielding at temperatures up to room temperature; the maximum and yield loads are the same. Heat 69 shows strain hardening at all temperatures and fractures at higher loads, although the ferrite contents of the two heats are comparable. Failure occurs at  $\approx 16$  kN load in Heat 68 and  $\approx 20$  kN load in Heat 69. The difference in the maximum loads at failure suggests a difference in fracture mechanism.

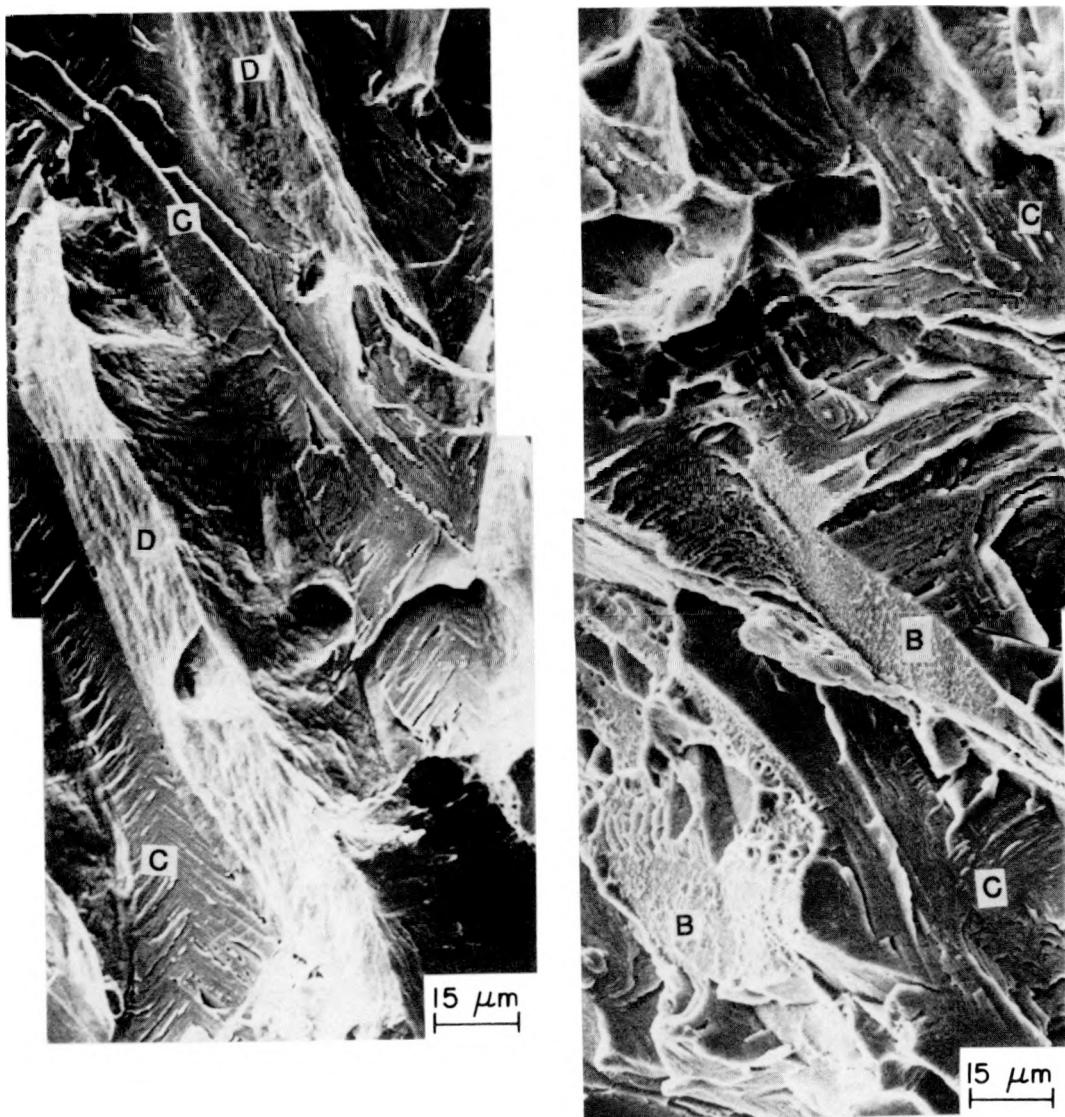


Figure 1. Fracture surfaces of Heat 68 aged for 10,000 h at 400°C and tested at room temperature. Fracture mode B = phase-boundary separation, C = cleavage of ferrite, D = ductile failure.

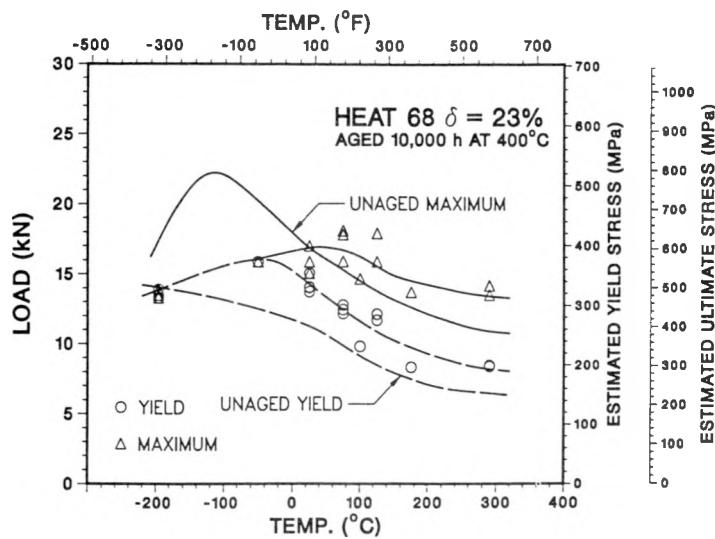
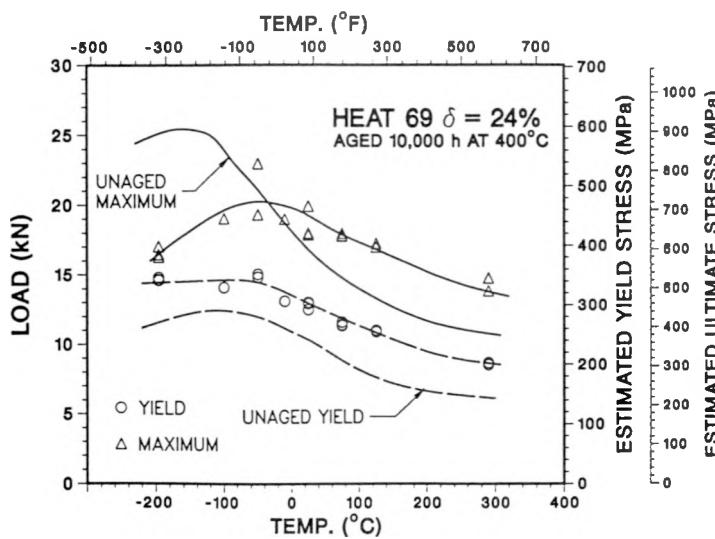


Figure 2. Yield and maximum loads from Charpy-impact tests for Heats 68 and 69, aged for 10,000 h at 400°C



The effects of material variables on the embrittlement of cast stainless steels have been evaluated. The kinetics and extent of embrittlement are controlled by several mechanisms that depend on material parameters and aging temperature. Materials aged at 450°C show significant precipitation of phase-boundary carbides (also nitrides in high-N steels) and a large decrease in ferrite content.<sup>10,12</sup> At reactor temperatures, such processes either do not occur or their kinetics are extremely slow. Consequently, data obtained at 450°C aging do not reflect reactor operating conditions, and extrapolation of these data to predict the extent of embrittlement at reactor temperatures is not valid. The chemical composition of the steel and the ferrite content and spacing are important parameters in controlling the extent and kinetics of embrittlement. Ferrite morphology strongly affects the extent of embrittlement, whereas material composition influences the kinetics of embrittlement. Small changes in the constituent elements of the cast material can cause the kinetics of embrittlement to vary significantly. The kinetics of embrittlement are controlled by spinodal decomposition and precipitation of G phase in ferrite, as well as by precipitation and

growth of phase-boundary carbides. The rate of embrittlement for a specific cast stainless steel depends on the relative contributions of carbide and G-phase precipitation; activation energies can range from 65 to 230 kJ/mole. An initial assessment of the mechanisms and the significance of low-temperature embrittlement of cast stainless steels in LWR systems have been presented.<sup>12</sup>

This report presents Charpy-impact data on several heats of cast stainless steel aged up to 30,000 h at 320, 350, and 400°C. The results are analyzed to establish the kinetics of embrittlement and estimate the extent of embrittlement of cast stainless steels from known material parameters.

## 2 Mechanical Properties

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### 2.1 Room-Temperature Impact Energy

The Charpy-impact data for various experimental and commercial heats, aged up to 30,000 h at 290, 320, 350, 400, and 450°C, were presented in Ref. 12. The results from room-temperature Charpy-impact tests were analyzed to determine the kinetics and extent of embrittlement. The variation of Charpy-impact energy  $C_v$  (J/cm<sup>2</sup>) with time can be expressed as

$$\log_{10}C_v = \log_{10}C_{vsat} + \beta\{1 - \tanh \{[(P - \theta)/\alpha]\}\}, \quad (1)$$

where  $P$  is the aging parameter,  $C_{vsat}$  (J/cm<sup>2</sup>) is the minimum impact energy reached after long-term aging,  $\beta$  is half the maximum decrease in impact energy,  $\theta$  is the log of the time to achieve  $\beta$  reduction in impact energy, and  $\alpha$  is a shape factor. The aging parameter represents the time to achieve a specific degree of aging at 400°C. The aging time to reach a given degree of embrittlement at different temperatures is determined from the equation

$$t = 10^P \exp\left[\frac{Q}{R}\left(\frac{1}{T} - \frac{1}{673}\right)\right], \quad (2)$$

where  $Q$  is the activation energy,  $R$  is the gas constant,  $T$  is the absolute temperature, and  $P$  is the aging parameter that represents the degree of aging reached after  $10^P$  h at 400°C.

The mechanical-property data indicate that the kinetics and extent of embrittlement are controlled by several mechanisms that depend on material parameters and aging temperature. Precipitation of phase-boundary carbides and/or growth of existing carbides occurs in CF-8 and CF-8M steels during aging at 400 and 450°C. The fracture surfaces of Charpy-impact specimens of Heat 60, unaged and aged at 400 and 450°C and tested at -197°C, are shown in Fig. 3. The fracture mode of the specimen aged at 450°C is predominantly phase-boundary separation. Furthermore, thermal aging at 450°C leads to substantial decrease in ferrite content of the steel (Fig. 4). At reactor temperatures, such processes either do not occur or their kinetics are extremely slow. Because the data obtained after 450°C aging are not representative of reactor operating conditions, they were excluded from the analysis. The Charpy data obtained after 290°C aging showed no reduction in impact energy even after aging for 30,000 h; instead, a slight increase in impact energy was observed relative to the unaged material. This increase in impact energy is real and was

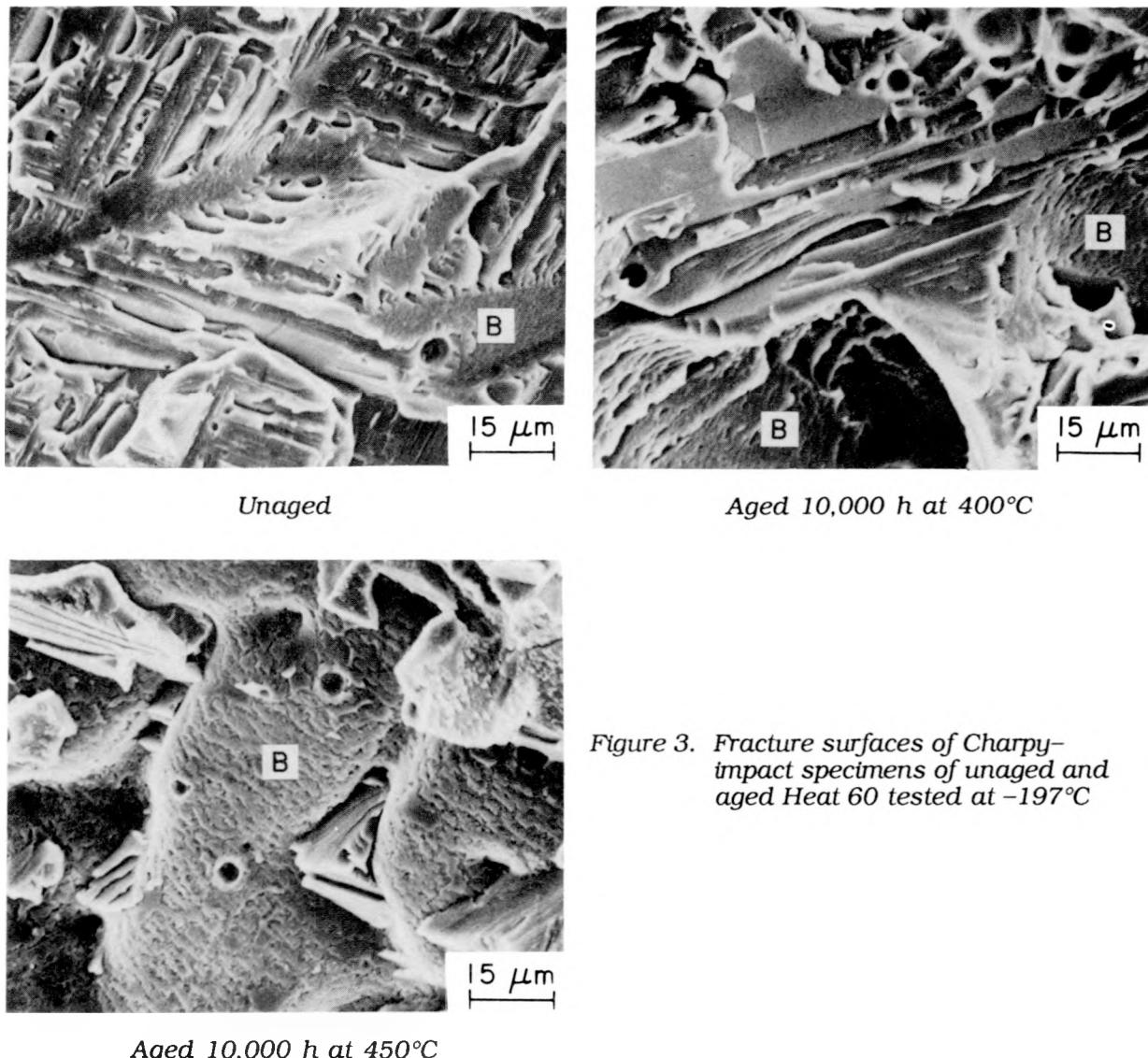


Figure 3. Fracture surfaces of Charpy-impact specimens of unaged and aged Heat 60 tested at  $-197^{\circ}\text{C}$

observed for most heats aged at low temperatures, i.e., aged up to 30,000 h at  $290^{\circ}\text{C}$  or up to 10,000 h at  $320^{\circ}\text{C}$ . The data from the relatively short-time aging at  $290^{\circ}\text{C}$  tend to bias the analyses to yield higher values of activation energies; therefore, the  $290^{\circ}\text{C}$  aging results were also excluded from the analysis.

The values of the constants in Eqs. 1 and 2 were obtained from best fit of the Charpy data for various heats of cast stainless steel aged up to 30,000 h at 320, 350, and  $400^{\circ}\text{C}$ . Activation energies, with the 95% confidence limits, are given in Table 2. The confidence limits are large for some heats because of the relatively small decrease in impact energy and large scatter in the data. The Charpy-impact data for three heats are plotted as a function of aging time in Fig. 5, along with the fitted curves determined from Eqs. 1 and 2.

The activation energies reported earlier<sup>10,12</sup> primarily represent the kinetics of embrittlement at temperatures between  $400$  and  $350^{\circ}\text{C}$ . Charpy data for long-term aging (i.e., 30,000 h) at  $320^{\circ}\text{C}$  were not included in the analyses. The earlier values are 15–20% lower than those determined from aging data at temperatures between  $400$  and  $320^{\circ}\text{C}$ . The

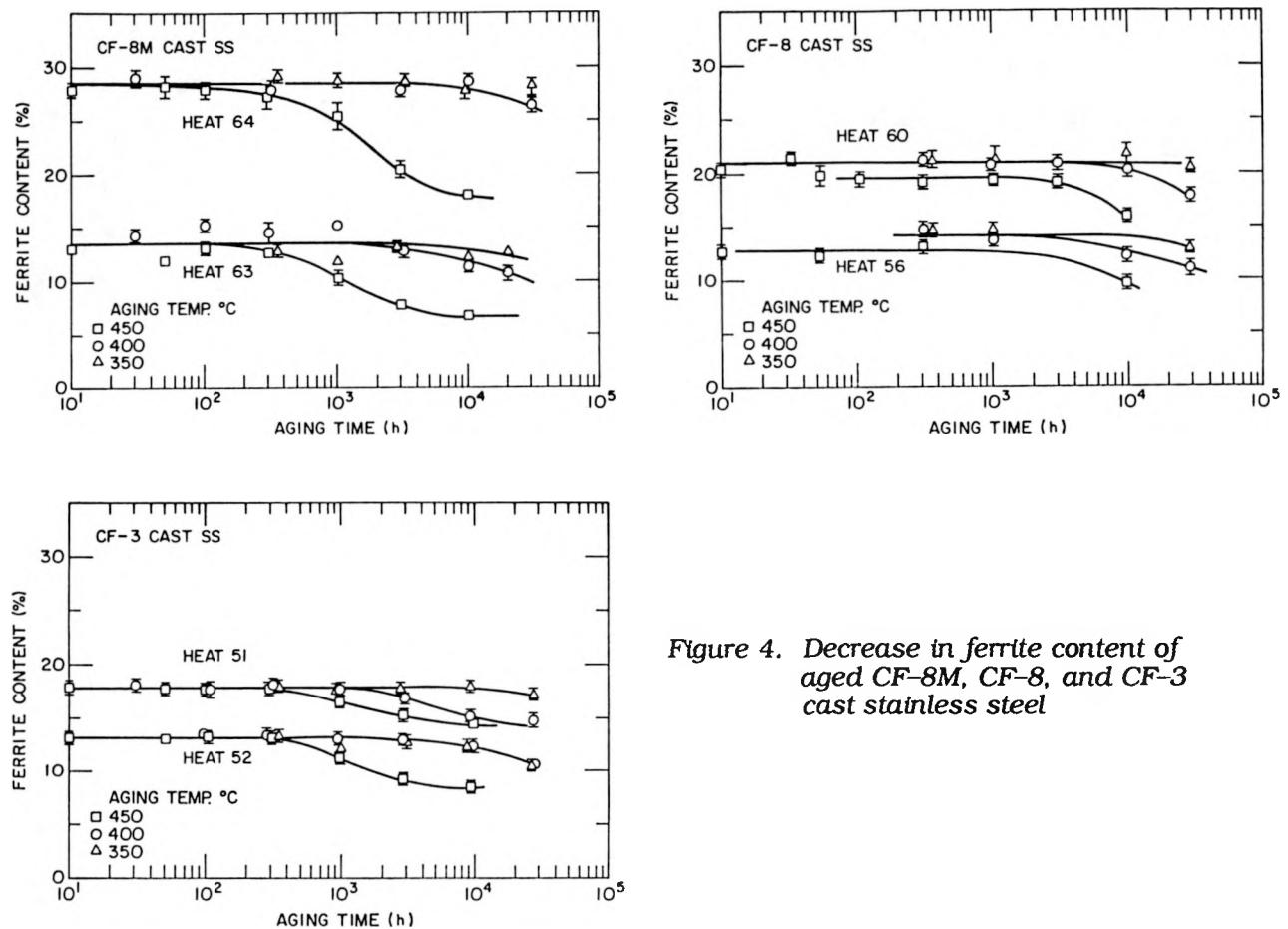


Figure 4. Decrease in ferrite content of aged CF-8M, CF-8, and CF-3 cast stainless steel

Table 2. Activation energies for kinetics of embrittlement of cast stainless steels

| Heat | Parameter | CV <sub>sat</sub><br>(J/cm <sup>2</sup> ) | Constants |          |          | Q {kJ/mole (kcal/mole)} |                       |
|------|-----------|---|-----------|----------|----------|-------------------------|-----------------------|
|      |           |   | $\beta$   | $\theta$ | $\alpha$ | Average                 | 95% Confidence Limit  |
| 47   | $\Phi^a$  | 174.2                                     | 0.063     | 2.35     | 1.40     | 187 (44.7)              | 73-300 (17.5-71.8) b  |
| 51   |           | 149.2                                     | 0.083     | 3.00     | 0.76     | 221 (52.8)              | 123-320 (29.3-76.4) b |
| 69   |           | 96.9                                      | 0.202     | 3.05     | 0.93     | 167 (40.0)              | 120-215 (28.7-51.3) b |
| 59   |           | 99.8                                      | 0.166     | 3.12     | 1.40     | 229 (54.7)              | 156-301 (37.4-72.0)   |
| 60   |           | 52.0                                      | 0.288     | 2.95     | 0.89     | 227 (54.2)              | 186-267 (44.4-63.9)   |
| 68   |           | 46.4                                      | 0.348     | 3.00     | 0.74     | 169 (40.5)              | 136-204 (32.4-48.2)   |
| P1   |           | 58.7                                      | 0.282     | 2.38     | 0.75     | 249 (59.6)              | 210-289 (50.2-69.1)   |
| 63   |           | 111.7                                     | 0.155     | 3.20     | 1.40     | 119 (28.4)              | 67-170 (16.0-40.7)    |
| 64   |           | 45.2                                      | 0.304     | 2.75     | 0.62     | 156 (37.4)              | 131-181 (31.4-43.2)   |
| 65   |           | 58.5                                      | 0.269     | 2.93     | 0.94     | 191 (45.7)              | 154-228 (36.8-54.6)   |
| 66   |           | 106.3                                     | 0.149     | 3.02     | 1.30     | 203 (48.4)              | 125-280 (29.9-66.9) b |
| 75   |           | 34.7                                      | 0.422     | 2.76     | 0.53     | 146 (34.8)              | 127-165 (30.3-39.4)   |
| P4   |           | 53.8                                      | 0.325     | 2.95     | 0.89     | 143 (34.2)              | 115-171 (27.6-40.8)   |

a Calculated from Eq. 4.

b Standard deviation is large because of the relatively small decrease in impact energy and a large scatter in data.

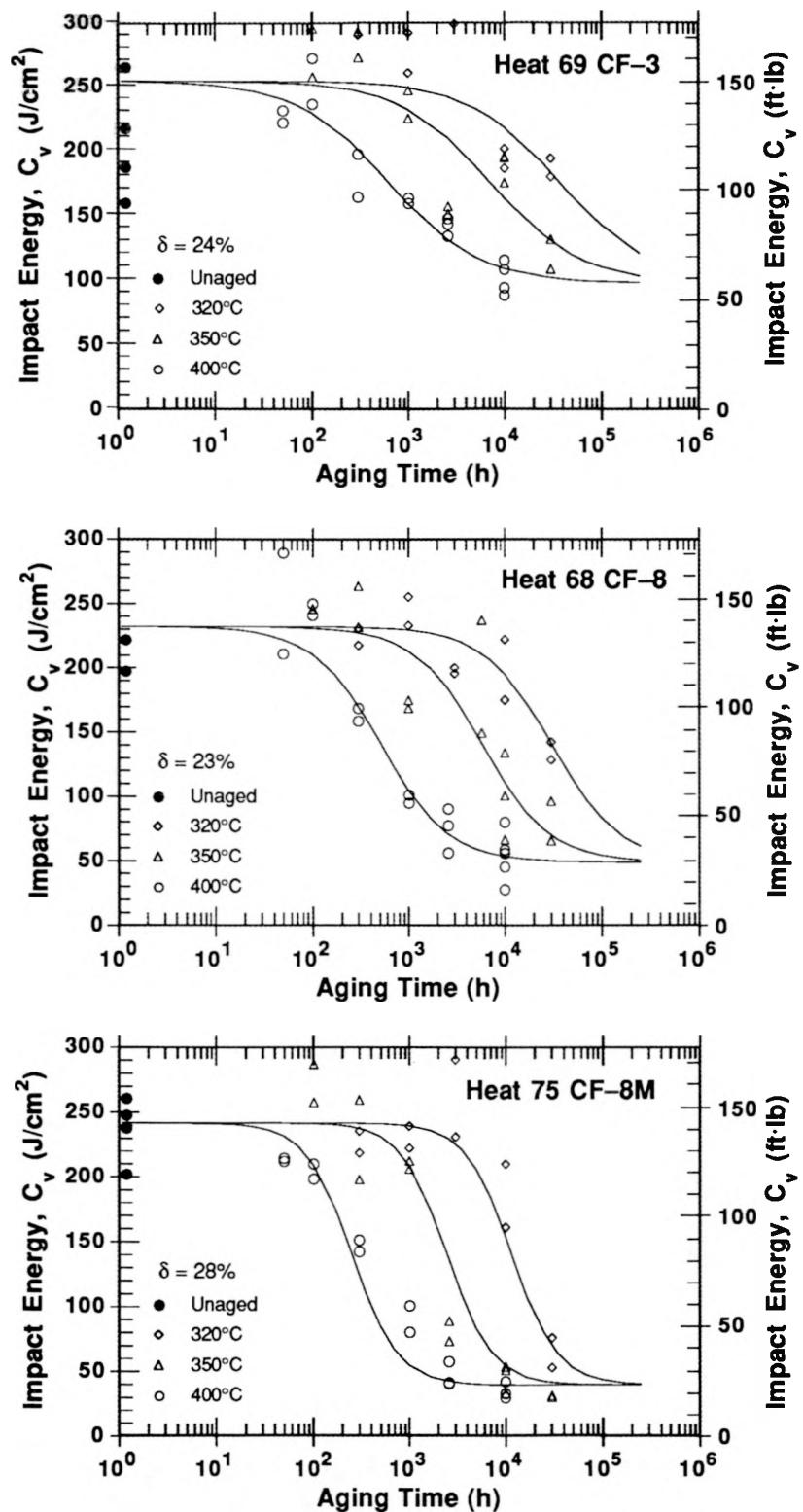


Figure 5. Effect of thermal aging on room-temperature impact energy of CF-3, CF-8, and CF-8M cast stainless steel

results suggest that the activation energy for embrittlement is not constant in the temperature range of 290–400°C, but increases with decrease in temperature. Similar behavior has been reported for several heats of CF-8M steels aged at temperatures between 300 and 400°C.<sup>16</sup> Consequently, extrapolation of the 400°C data to reactor temperatures by applying activation energies obtained from 350–400°C aging would yield conservative estimates of embrittlement for some of the heats of cast stainless steel.

## 2.2 Extent of Embrittlement

Charpy-impact data obtained at room temperature were analyzed to develop a correlation between material variables and the extent or degree of embrittlement, i.e., the minimum room-temperature impact energy,  $C_{Vsat}$  in Eq. 1, that could be achieved after long-term aging. It is well established that the extent of embrittlement increases with an increase in the ferrite content of cast stainless steel. Furthermore, Charpy-impact data for several heats of CF-8 and CF-8M steels aged for 10,000 h at 350 or 400°C indicate that the impact energy decreases with an increase in the Cr content, irrespective of the ferrite content in the steel.<sup>16</sup> A better correlation was obtained when the total concentration of ferrite formers (i.e., Cr, Mo, and Si) was considered.<sup>16</sup> A sharp decrease in impact energy occurs when either the Cr content exceeds 18 wt.% or the concentration of Cr+Mo+Si exceeds 23.5 wt.%. An increase in the concentration of C or N in the steel also increases the extent of embrittlement because of the contribution to phase-boundary carbides or nitrides and the subsequent fracture by phase-boundary separation. The influence of C content on the extent of embrittlement is clearly seen in Fig. 5. Although the ferrite content and concentrations of Cr+Mo+Si are comparable for Heats 68 and 69, the minimum impact energy for Heat 68 (CF-8) is significantly lower than that for Heat 69 (CF-3).

The data on the minimum room-temperature impact energy  $C_{Vsat}$  (J/cm<sup>2</sup>) were analyzed with an exponential function of the various material variables. The best fit of the data was obtained with the expression

$$\log_{10}C_{Vsat} = 1.386 + 0.938\exp(-0.0205\Phi). \quad (3)$$

The material parameter  $\Phi$  is given by

$$\Phi = \delta_m^2(Cr + Mo + Si)(C + 0.4N)Ni\lambda / 10^4, \quad (4)$$

where  $\delta_m$ , the measured ferrite content, is in %, the concentrations of Cr, Mo, Si, C, and N are in wt.%, and the mean ferrite spacing  $\lambda$  is in  $\mu\text{m}$ . A similar correlation, but without the effect of ferrite spacing, was proposed earlier by investigators at Electricité de France (EdF).\*

Minimum impact energy is plotted as a function of the material parameter  $\Phi$  in Fig. 6. Results from the studies at Framatome (FRA),<sup>17,18</sup> Central Electricity Generating Board (CEGB),<sup>19</sup> and Electric Power Research Institute (EPRI)<sup>20</sup> are also shown in the figure. The data show good correlation with the material parameter. However, the impact energy for

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\*M. Guttmann, EdF, private communication, October 1987.

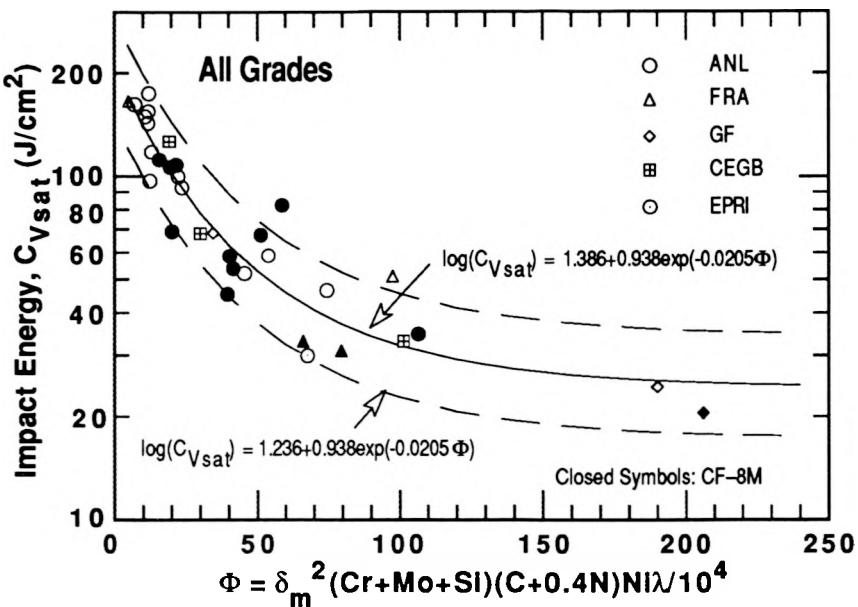


Figure 6. Correlation between minimum room-temperature impact energy and material parameter  $\Phi$  for aged cast stainless steel. The curves shown by dashed lines represent  $\pm 41\%$  deviation from predicted values.

FRA Heat 4331 (not shown), which contains 0.2 wt.% Nb, was significantly lower than that predicted by Eq. 3. The fracture surface of the impact test specimen of FRA Heat 4331 (Fig. 7) shows large Nb carbides at the phase boundaries. The presence of phase-boundary carbides alters the deformation and fracture behavior of the material, i.e., cleavage can be initiated by carbide cracking. A difference in the mode of fracture is reflected in the values of the yield and maximum loads for the instrumented Charpy tests, shown in Fig. 8 for FRA Heat 4331 and ANL Heat 74. The corresponding Charpy transition curves are shown in Fig. 9. Both heats are CF-8M grade and contain  $\sim 20\%$  ferrite, yet the transition curves are significantly different. The 65-J transition temperature is  $220^\circ\text{C}$  for Heat 4331, aged for 700 h at  $400^\circ\text{C}$ , and is  $20^\circ\text{C}$  for Heat 74 aged for 10,000 h at  $400^\circ\text{C}$ . The yield loads are

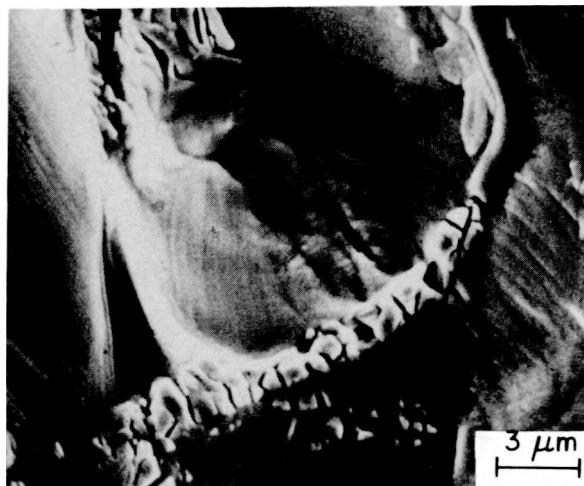


Figure 7. Fracture surface of Charpy-impact specimen of Framatome Heat 4331, aged for 700 h at  $400^\circ\text{C}$

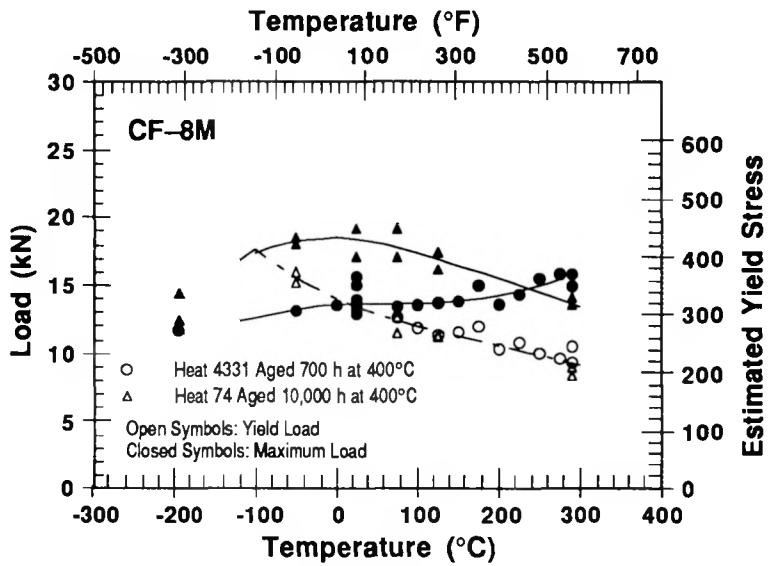


Figure 8. Yield and maximum loads from Charpy-impact data for Framatome Heat 4331 and ANL Heat 74

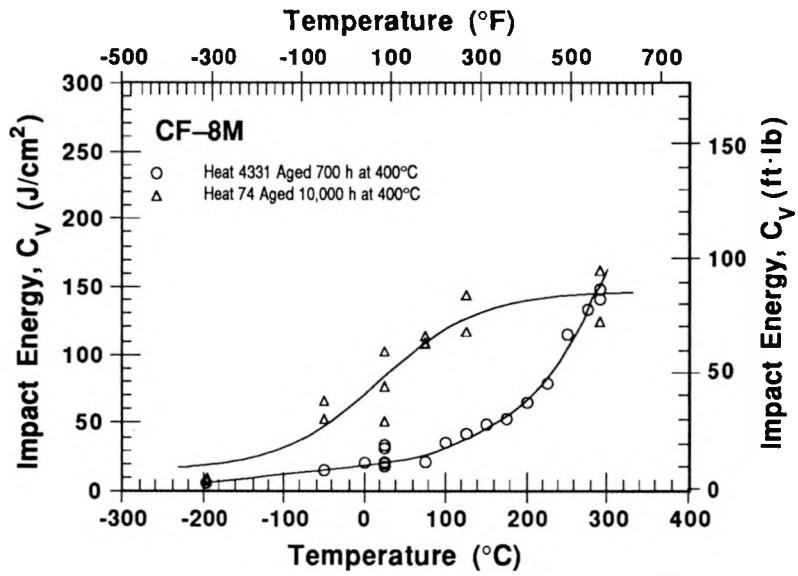


Figure 9. Charpy transition curves for Heats 4331 and 74 aged at 400°C

comparable for both heats, which indicates a similar degree of strengthening in the two heats. However, Fig. 8 shows that failure occurs at  $\approx 14$  kN load in Heat 4331 and at  $\approx 18$  kN load in Heat 74. The difference in the maximum load at failure suggests a difference in the fracture mechanism. The present correlation does not consider the effects of Nb on embrittlement.

The correlation in Fig. 6 indicates that the impact energy will be less than  $50$  J/cm $^2$  for cast stainless steels in which the material parameter is greater than  $\approx 50$ . For cast stainless steels containing  $>10\%$  ferrite, mean ferrite spacing is in the range of  $40$ – $200$   $\mu\text{m}$ ,

Cr+Mo+Si concentration is  $\approx 22$  wt.% for CF-8 or CF-3 and  $\approx 24$  wt.% for CF-8M, and N content is typically 0.04 wt.%. Thus, for cast materials with 0.06 wt.% C, 9 wt.% Ni, and 100  $\mu\text{m}$  ferrite spacing, impact energy will be below 50 J/cm<sup>2</sup> when the ferrite content is above 18%. However, cast materials with 10 or 15% ferrite can also reach very low impact strengths with an increase in any one of the variables included in the material parameter  $\Phi$ . For example, Ni content is often  $> 10$  wt.% for CF-8M steels and values of  $\lambda$  as high as 200  $\mu\text{m}$  have been observed for several heats of cast stainless steel (Table 1).

Equations 3 and 4 can be used to estimate the extent of embrittlement at saturation (expressed in terms of room-temperature impact energy) for a specific cast stainless steel component. The variables in the material parameter can be determined nondestructively. The compositions are generally known, and ferrite content can be calculated from the composition or measured with a magne-gage or ferrite scope. Ferrite spacing is the variable that is least readily available, but it can be determined by surface replica techniques. However, a conservative estimate of the possible extent of embrittlement can be obtained by assuming the highest value observed in the material data base, i.e., a spacing of  $\approx 200$   $\mu\text{m}$ .

The effect of material parameter  $\Phi$  on the extent of embrittlement is clearly seen in Fig. 10. The impact energies for several heats with comparable values of  $\Phi$  are plotted as a function of the aging parameter  $P$ . The different temperatures and times of aging are normalized in terms of  $P$  by Eq. 2. The results show that the extent of embrittlement increases with an increase in  $\Phi$ . For each range of  $\Phi$  there is a saturation value of minimum impact energy. This behavior is independent of the kinetics of embrittlement, e.g., the extent of embrittlement is comparable for Heats 60, P1, 64, 65, and P4, whereas the activation energies range from 140 to 240 kJ/mole. Ferrite content also varies significantly for these heats: 10% for Heat P4 and 28% for Heat 64.

## 2.3 Charpy Transition Curves

Assessment of the changes in impact strength at reactor operating temperatures requires evaluation of aging effects on Charpy transition curves. The Charpy data were fitted with a hyperbolic tangent function of the form

$$C_V = K_0 + B \{1 + \tanh [(T - C)/D]\}, \quad (5)$$

where  $K_0$  is the lower-shelf energy,  $T$  is the test temperature,  $B$  is half the distance between upper-shelf energy (USE) and lower-shelf energy,  $C$  is the mid-shelf Charpy transition temperature (CTT) in  $^{\circ}\text{C}$ , and  $D$  is the half-width of the transition region. The values of the constants in Eq. 5 are given in Table 3. The effect of aging time on the transition curves for the three grades of cast stainless steel is shown in Fig. 11. The effect of aging temperature for cast materials that were aged for 30,000 h is shown in Fig. 12. Thermal aging decreases the impact energy and shifts the transition curves to higher temperatures. The results indicate a "saturation effect" for USE after aging. The values of USE decrease significantly during aging for  $\approx 2600$  h at  $350^{\circ}\text{C}$  but do not decrease further with longer aging times. After saturation of USE, the decrease in room-temperature impact energy is due to an increase in CTT, i.e., the value of  $B$  in Eq. 5 remains constant and only  $C$  and  $D$  increase with longer aging times. This behavior is observed at all aging temperatures and for all heats of material. The three heats that were studied contain similar amounts of ferrite,

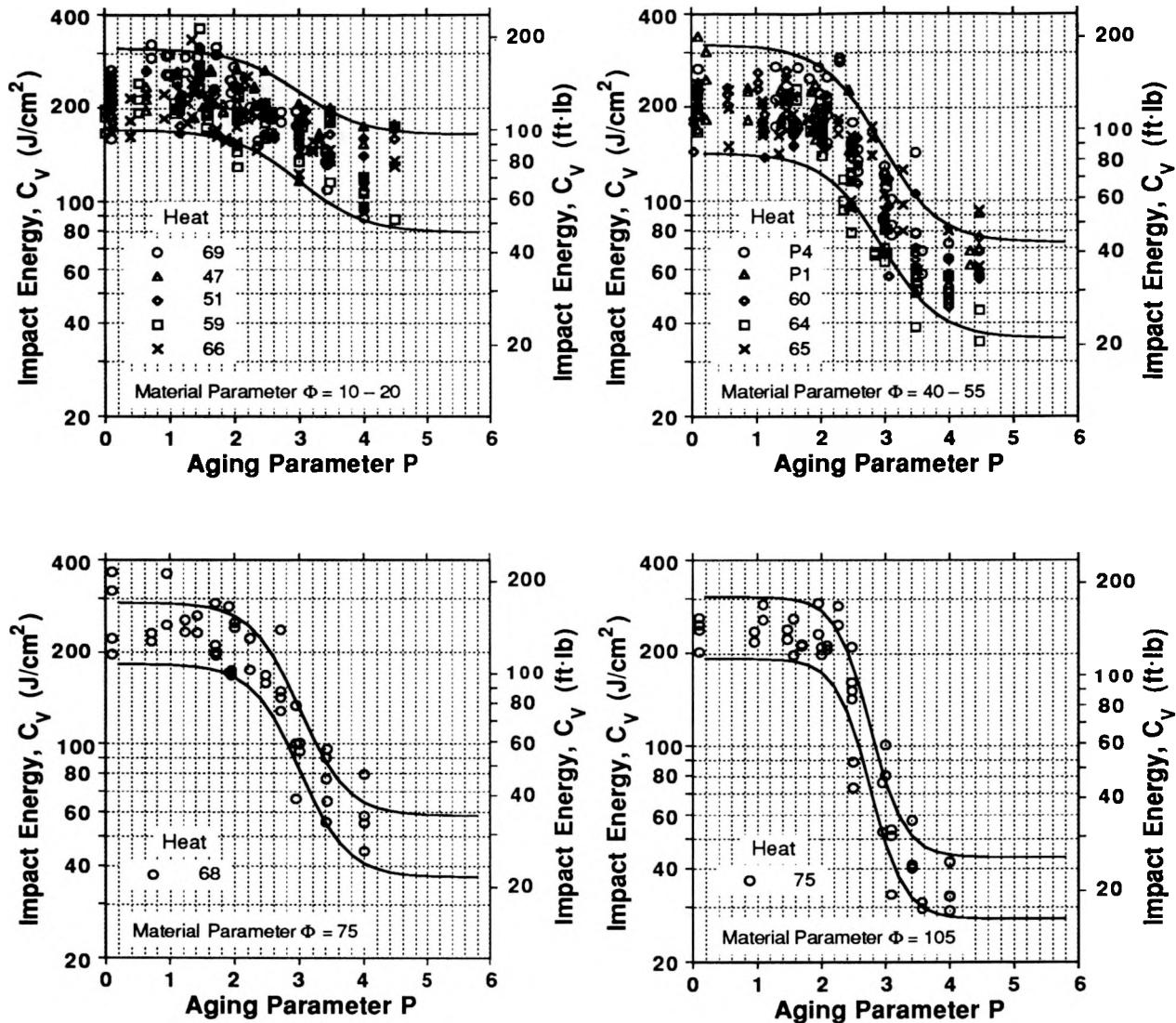


Figure 10. Effect of thermal aging on room-temperature impact energy of CF-3, CF-8, and CF-8M steels with various material parameters  $\Phi$

and the saturation values of USE are comparable. However, the shift in CTT is significantly different: the CF-8M steel exhibits the largest shift.

The room-temperature Charpy data (Fig. 5) indicate that for Heats 68, 69, and 75 of cast stainless steel, a saturation value of minimum impact energy is reached after  $\approx 3,000$  h at  $400^\circ\text{C}$  or  $\approx 30,000$  h at  $350^\circ\text{C}$ , i.e., when the aging parameter  $P$  is  $\approx 3.5$ . Because both room-temperature impact energy and USE appear to reach saturation values, Charpy transition curves may also show a "saturation effect." Transition curves for three grades of cast stainless steel aged to  $P$  values of 3.4–4.0 are shown in Fig. 13. For Heat 69 ( $\Phi \approx 12$ ), the transition curves for the three aging conditions are comparable. The minimum room-temperature impact energy is close to USE. This suggests a saturation effect on the transition curve, because an increase in CTT would mean a further decrease in room-temperature impact energy. Thus, the curves represent the highest CTT that would be achieved after

Table 3. Values of constants in Eq. 5 for Charpy transition curve of cast stainless steels

| Heat | Aging Condition |          | Constants                           |                        |        |        |
|------|-----------------|----------|-------------------------------------|------------------------|--------|--------|
|      | Temp. (°C)      | Time (h) | K <sub>0</sub> (J/cm <sup>2</sup> ) | B (J/cm <sup>2</sup> ) | C (°C) | D (°C) |
| 69   | Unaged          | -        | 40                                  | 130.3                  | -186.8 | 222.7  |
|      | 290             | 30,000   |                                     | 86.4                   | -176.8 | 30.0   |
|      | 320             | 10,000   |                                     | 78.2                   | -154.1 | 37.8   |
|      | 320             | 30,000   |                                     | 90.5                   | -74.2  | 89.2   |
|      | 350             | 2,570    |                                     | 69.1                   | -113.2 | 50.4   |
|      | 350             | 10,000   |                                     | 73.5                   | -34.6  | 43.5   |
|      | 350             | 30,000   |                                     | 59.2                   | -16.9  | 107.1  |
|      | 400             | 2,570    |                                     | 55.6                   | -60.7  | 87.4   |
|      | 400             | 10,000   |                                     | 54.7                   | -16.9  | 98.0   |
|      | 450             | 2,570    |                                     | 50.6                   | -85.0  | 126.5  |
| I    | Unaged          | -        | 50                                  | 62.8                   | -307.9 | 106.2  |
|      | 320             | 30,000   |                                     | 81.8                   | -184.0 | 127.9  |
|      | 350             | 9,980    |                                     | 49.8                   | -125.4 | 51.7   |
|      | 400             | 9,980    |                                     | 44.3                   | -120.9 | 70.4   |
| P2   | Unaged          | -        | 50                                  | 162.1                  | -189.0 | 44.1   |
|      | 320             | 30,000   |                                     | 172.7                  | -42.2  | 71.9   |
|      | 350             | 10,000   |                                     | 148.7                  | -43.1  | 87.8   |
|      | 350             | 30,000   |                                     | 157.0                  | -5.8   | 97.6   |
| 68   | Unaged          | -        | 15                                  | 138.3                  | -59.8  | 99.9   |
|      | 290             | 30,000   |                                     | 169.0                  | -12.1  | 98.4   |
|      | 320             | 10,000   |                                     | 97.4                   | -40.9  | 46.0   |
|      | 320             | 30,000   |                                     | 110.1                  | -0.9   | 121.2  |
|      | 350             | 5,780    |                                     | 78.5                   | -12.6  | 60.7   |
|      | 350             | 10,000   |                                     | 97.0                   | 28.0   | 67.3   |
|      | 350             | 30,000   |                                     | 86.3                   | 53.3   | 85.7   |
|      | 400             | 2,570    |                                     | 70.5                   | 31.3   | 78.5   |
|      | 400             | 10,000   |                                     | 68.6                   | 64.0   | 64.3   |
|      | 450             | 2,570    |                                     | 54.9                   | 32.6   | 86.7   |
| 70   | Unaged          | -        | 15                                  | 119.7                  | -156.2 | 60.6   |
|      | 350             | 2,570    |                                     | 105.6                  | -77.0  | 38.7   |
|      | 350             | 10,000   |                                     | 89.2                   | 23.6   | 121.7  |
|      | 400             | 2,570    |                                     | 87.4                   | -1.7   | 116.2  |
|      | 400             | 10,000   |                                     | 71.2                   | 10.3   | 90.9   |
| 74   | Unaged          | -        | 15                                  | 89.5                   | -177.5 | 119.6  |
|      | 290             | 30,000   |                                     | 140.9                  | -104.8 | 40.7   |
|      | 320             | 10,000   |                                     | 91.1                   | -95.0  | 65.8   |
|      | 320             | 30,000   |                                     | 87.1                   | -8.5   | 84.3   |
|      | 350             | 2,570    |                                     | 89.0                   | -88.7  | 38.9   |
|      | 350             | 10,000   |                                     | 71.5                   | -35.6  | 86.7   |
|      | 350             | 30,000   |                                     | 81.2                   | 79.4   | 168.5  |
|      | 400             | 2,570    |                                     | 61.6                   | -37.2  | 49.6   |
|      | 400             | 10,000   |                                     | 65.6                   | 19.2   | 123.7  |
|      | 450             | 2,570    |                                     | 39.2                   | -47.0  | 63.5   |
| 75   | Unaged          | -        | 15                                  | 92.0                   | -156.5 | 43.7   |
|      | 290             | 30,000   |                                     | 121.6                  | -76.4  | 50.5   |
|      | 320             | 10,000   |                                     | 90.4                   | -16.0  | 37.9   |
|      | 320             | 30,000   |                                     | 78.4                   | 83.0   | 140.1  |
|      | 350             | 2,570    |                                     | 70.9                   | -10.2  | 105.2  |
|      | 350             | 10,000   |                                     | 71.3                   | 80.5   | 74.4   |
|      | 350             | 30,000   |                                     | 67.9                   | 207.7  | 180.1  |
|      | 400             | 2,570    |                                     | 52.7                   | 45.5   | 83.1   |
|      | 400             | 10,000   |                                     | 66.3                   | 140.7  | 138.8  |
|      | 50              | 2,570    |                                     | 34.0                   | 20.8   | 99.4   |

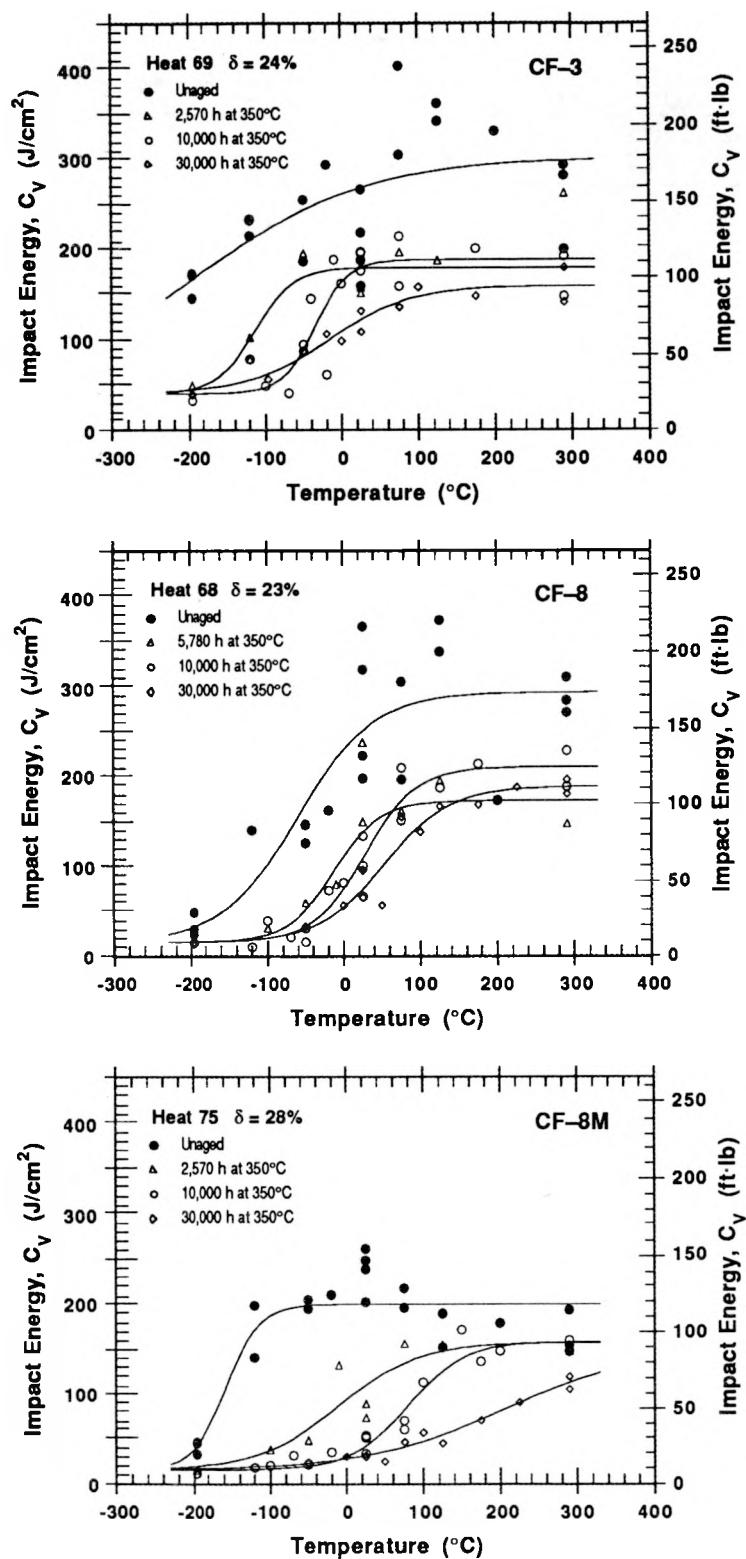


Figure 11. Effect of aging time on Charpy transition curves of CF-3, CF-8, and CF-8M steels aged at 350°C

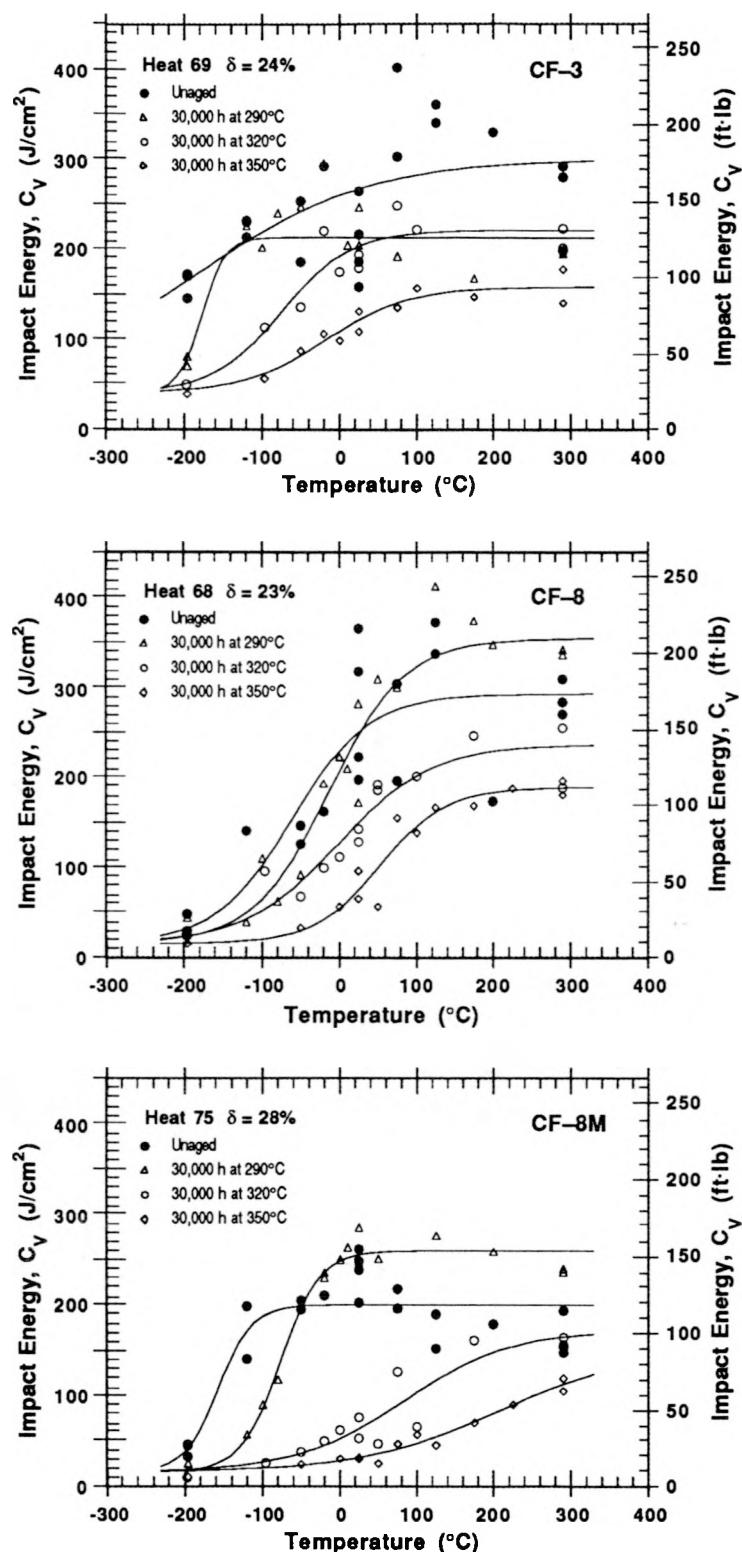


Figure 12. Effect of temperature on Charpy transition curves of CF-3, CF-8, and CF-8M steels aged for 30,000 h

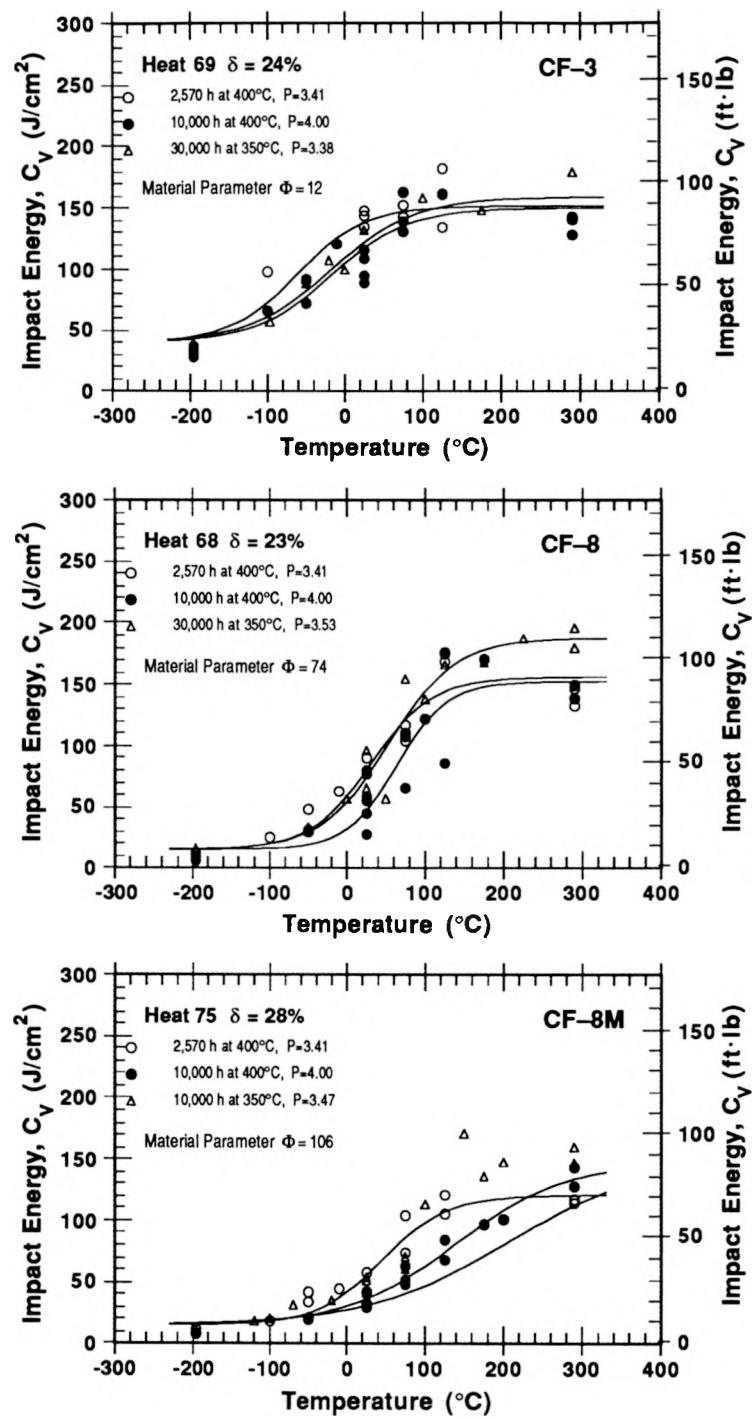


Figure 13. Charpy transition curves for CF-3, CF-8, and CF-8M steels aged to  $P$  values between 3.4 and 4.0

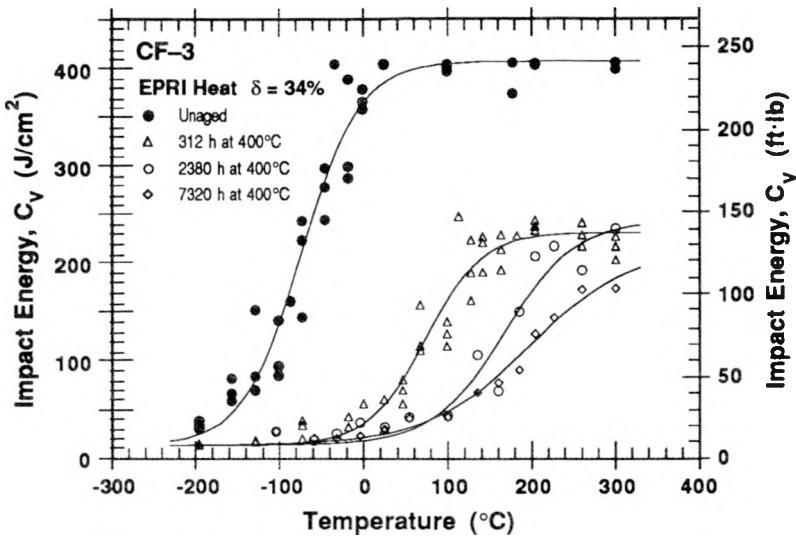


Figure 14. Charpy transition curves for CF-3 cast stainless steel aged at 400°C for various times. Charpy-impact data from Ref. 20.

long-term aging. The transition curves for Heat 68 ( $\Phi \approx 74$ ) are also comparable but may not represent saturation. Because the minimum room-temperature impact energy is close to the lower-shelf energy, an increase in CTT would decrease the impact energy at reactor temperatures without significantly changing room-temperature impact energy. The results for Heat 75 ( $\Phi \approx 106$ ) do not indicate a saturation effect on the transition curve. Thus, although the room-temperature impact energy does not change for  $P$  values  $>3.5$ , the impact energy at reactor temperatures may continue to decrease with time. This behavior is not unique to CF-8M steels and was observed in the EPRI study<sup>20</sup> on CF-3 steel ( $\Phi \approx 68$ ), as shown in Fig. 14. Long-term aging data are needed to establish the impact energies at reactor temperatures and to confirm whether a saturation effect in transition curves is achieved for some of the heats, e.g., heats with low values of material parameter  $\Phi$ .

The Charpy data for Heats 68, 69, and 75 also indicate that the transition curves for 350°C aging are lower than those after aging for an equivalent time at 400°C, particularly for the CF-8M steel. The mid-shelf CTT for the three grades of cast stainless steel aged up to 30,000 h at 320, 350, 400, and 450°C are plotted as a function of the aging parameter  $P$  in Fig. 15. The increase in CTT (i.e., slope of the solid lines) for CF-8M steels is greater by a factor of two than that for CF-3 or CF-8 steels. For all heats, the values of CTT for 400 or 450°C aging are lower than those after aging at lower temperatures for equivalent times, i.e., for the same values of  $P$ . These results raise an important issue, i.e., the kinetics of embrittlement established from the room-temperature Charpy data are not representative of the changes in Charpy-impact energy at reactor temperatures. Also, the data for minimum Charpy-impact energy at 290°C show poor correlation with either the aging parameter  $P$  or the material parameter  $\Phi$ .

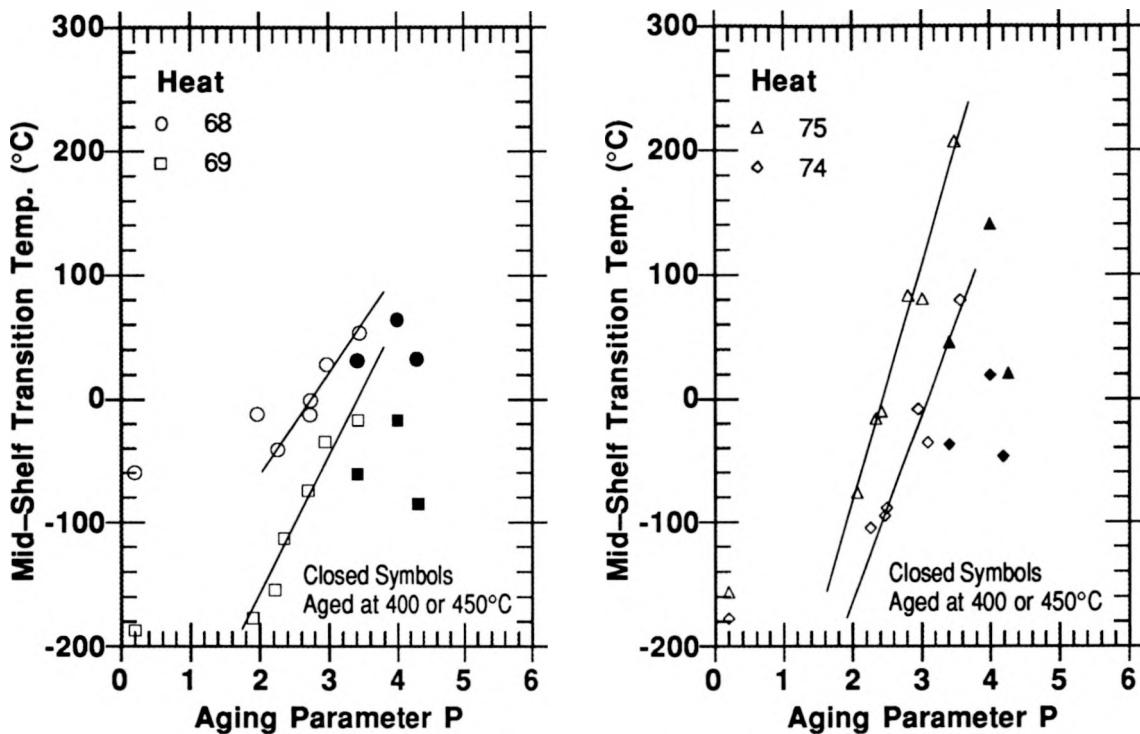
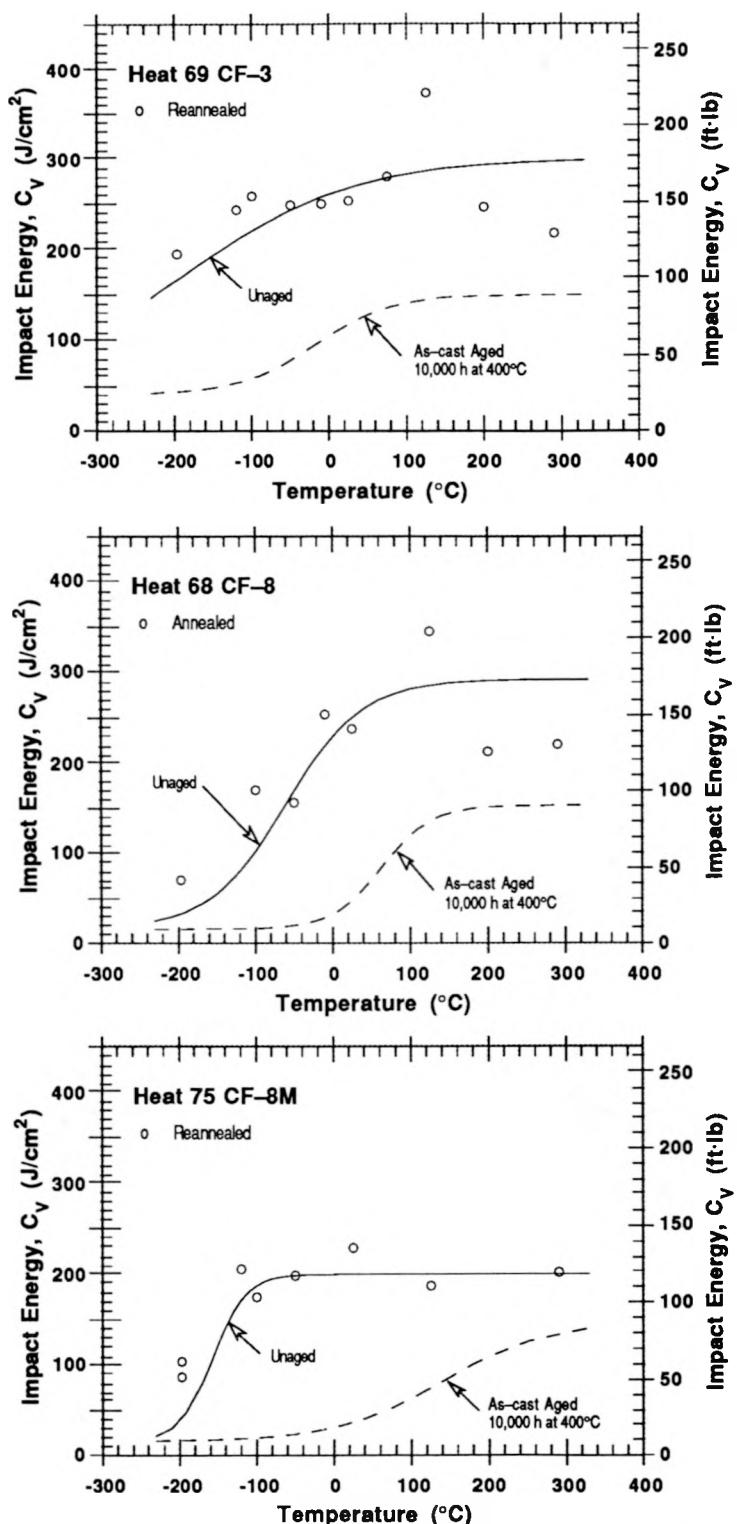


Figure 15. Change in mid-shelf CTT with thermal aging for CF-8 (Heat 68), CF-3 (Heat 69), and CF-8M (Heats 74 and 75) cast stainless steel. The solid lines represent the change in CTT with P for aging temperatures  $\leq 350^{\circ}\text{C}$ .

### 3 Recovery Annealing

Microstructural and annealing studies<sup>8,13-15</sup> on laboratory- and reactor-aged materials have been conducted to investigate the possibility of recovering the mechanical properties of embrittled materials. The results indicate that the formation of  $\alpha'$  phase by spinodal decomposition is the primary mechanism for embrittlement. The  $\alpha'$  phase is not stable at temperatures  $>550^{\circ}\text{C}$ . The mechanical properties can be recovered by annealing the embrittled cast stainless steels for 1 h at  $550^{\circ}\text{C}$  and water-quenching to dissolve the  $\alpha'$  phase while avoiding the formation of  $\sigma$  phase. The influence of annealing on the Charpy transition curves for three laboratory-aged heats and service-aged material from the KRB reactor is shown in Figs. 16 and 17, respectively. The service-aged pump cover plate was obtained from the KRB reactor, which was in service in Gundremmingen, West Germany, for  $\approx 12$  y (i.e.,  $\approx 8$  y, or  $\approx 68,000$  h, at a service temperature of  $284^{\circ}\text{C}$ ). The chemical composition and ferrite content of the KRB material are shown in Table 1. The results indicate essentially complete recovery from embrittlement; the transition curves for the annealed materials agree well with those for the unaged steel. Microstructural examination of the annealed material showed no  $\alpha'$  phase, but the size and distribution of the G phase were the same as in the aged material.<sup>13-15</sup>

Charpy data for laboratory-aged materials indicate that the kinetics of embrittlement can also be obtained from the reembrittlement of recovery-annealed material. The Charpy-



*Figure 16. Effect of annealing on the Charpy transition curves for thermally aged CF-3, CF-8, and CF-8M steel. Embrittled materials shown by dashed lines.*

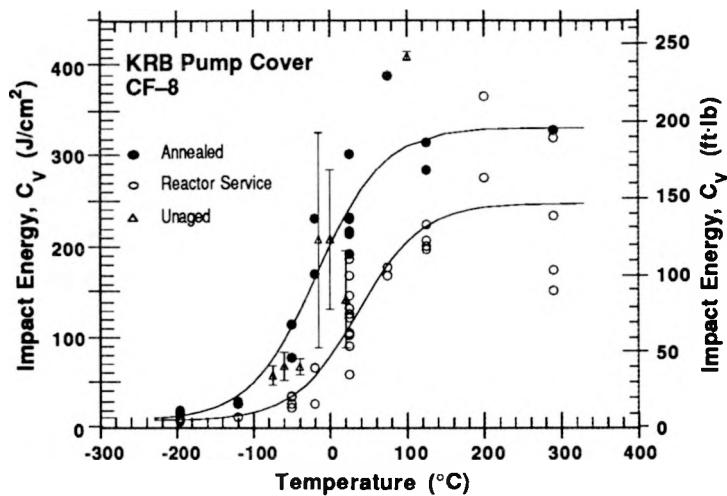


Figure 17. Effect of annealing on the Charpy transition curve for the KRB pump cover plate

impact data for recovery-annealed CF-3, CF-8, and CF-8M steels aged up to 10,000 h at 400, 350, and 320°C are given in Table 4. The Charpy transition data for the recovery-annealed materials aged for 10,000 h at 400°C are shown in Fig. 16. The transition curves are virtually the same as those for the as-cast material aged for 10,000 h at 400°C. The aging behaviors of as-cast and recovery-annealed steels are shown in Fig. 18. The activation energies of the as-cast materials are  $168 \pm 37$ ,  $167 \pm 48$ , and  $146 \pm 21$  kJ/mole for Heats 68, 69, and 75, respectively. The Charpy data for recovery-annealed and aged Heats 68, 69, and 75, respectively, yield activation energies of  $122 \pm 41$ ,  $176 \pm 49$ , and  $130 \pm 28$  kJ/mole, i.e., the kinetics of re-embrittlement of Heat 69 are comparable and those of Heats 68 and 75

Table 4. Charpy-impact test results for recovery-annealed and aged cast stainless steel

| Specimen Number <sup>a</sup> | Heat | Aging Temp. (°C) | Aging Time (h) | Test Temp. (°C) | Impact Energy (J/cm <sup>2</sup> ) | Yield Load (kN) | Max. Load (kN) |
|------------------------------|------|------------------|----------------|-----------------|------------------------------------|-----------------|----------------|
| CF-8 Grade                   |      |                  |                |                 |                                    |                 |                |
| 683-55A                      | 68   | —                | —              | -197            | 70.0                               | 15.838          | 19.607         |
| 683-54A                      | 68   | —                | —              | -50             | 155.3                              | 12.977          | 20.994         |
| 683-53A                      | 68   | —                | —              | 25              | 237.4                              | 10.946          | 16.785         |
| 683-52A                      | 68   | —                | —              | 125             | 345.0                              | 9.267           | 14.451         |
| 683-51A                      | 68   | —                | —              | 290             | 220.5                              | 7.382           | 11.883         |
| 684-52A                      | 68   | —                | —              | -100            | 169.6                              | 14.276          | 21.472         |
| 684-53A                      | 68   | —                | —              | -10             | 253.3                              | 11.893          | 18.572         |
| 684-54A                      | 68   | —                | —              | 200             | 212.7                              | 8.144           | 12.850         |
| 68-317A                      | 68   | 320              | 3,000          | 25              | 214.5                              | 10.770          | 16.024         |
| 68-318A                      | 68   | 320              | 3,000          | 25              | 184.3                              | 10.926          | 17.322         |
| 68-319A                      | 68   | 320              | 10,000         | 25              | 157.3                              | 12.010          | 18.455         |
| 68-320A                      | 68   | 320              | 10,000         | 25              | 208.0                              | 11.893          | 18.211         |
| 68-308A                      | 68   | 350              | 100            | 25              | 236.1                              | 11.327          | 17.683         |
| 68-309A                      | 68   | 350              | 300            | 25              | 230.9                              | 11.112          | 17.020         |
| 68-310A                      | 68   | 350              | 300            | 25              | 280.8                              | 11.883          | 17.615         |
| 68-311A                      | 68   | 350              | 1,000          | 25              | 160.8                              | 10.946          | 17.273         |

Table 4. (Contd.)

| Specimen Number <sup>a</sup> | Heat | Aging Temp. (°C) | Aging Time (h) | Test Temp. (°C) | Impact Energy (J/cm <sup>2</sup> ) | Yield Load (kN) | Max. Load (kN) |
|------------------------------|------|------------------|----------------|-----------------|------------------------------------|-----------------|----------------|
| 68-312A                      | 68   | 350              | 1,000          | 25              | 166.4                              | 10.809          | 17.527         |
| 68-313A                      | 68   | 350              | 3,000          | 25              | 84.1                               | 11.063          | 14.578         |
| 68-314A                      | 68   | 350              | 3,000          | 25              | 65.6                               | 11.239          | 14.061         |
| 68-315A                      | 68   | 350              | 10,000         | 25              | 55.2                               | 12.782          | 15.008         |
| 68-316A                      | 68   | 350              | 10,000         | 25              | 71.1                               | 12.352          | 16.063         |
| 684-58A                      | 68   | 400              | 50             | 25              | 213.7                              | 11.278          | 16.941         |
| 684-59A                      | 68   | 400              | 50             | 25              | 179.0                              | 11.249          | 16.600         |
| 68-301A                      | 68   | 400              | 100            | 25              | 211.5                              | 12.059          | 17.683         |
| 684-60A                      | 68   | 400              | 100            | 25              | 186.0                              | 11.756          | 17.635         |
| 68-302A                      | 68   | 400              | 300            | 25              | 175.9                              | 12.352          | 18.611         |
| 68-303A                      | 68   | 400              | 300            | 25              | 199.7                              | 11.835          | 18.347         |
| 68-304A                      | 68   | 400              | 1,000          | 25              | 126.5                              | 11.903          | 17.254         |
| 68-305A                      | 68   | 400              | 1,000          | 25              | 103.9                              | 12.791          | 16.287         |
| 68-306A                      | 68   | 400              | 3,000          | 25              | 73.7                               | 11.678          | 14.803         |
| 68-307A                      | 68   | 400              | 3,000          | 25              | 78.5                               | 11.962          | 14.783         |
| 683-56A                      | 68   | 400              | 10,000         | -197            | 12.1                               | 13.358          | 13.358         |
| 684-55A                      | 68   | 400              | 10,000         | -120            | 28.0                               | 16.277          | 16.277         |
| 683-57A                      | 68   | 400              | 10,000         | -50             | 32.3                               | 14.051          | 14.325         |
| 681-59A                      | 68   | 400              | 10,000         | 25              | 82.6                               | 13.368          | 16.063         |
| 682-59A                      | 68   | 400              | 10,000         | 25              | 66.7                               | 12.811          | 15.164         |
| 683-58A                      | 68   | 400              | 10,000         | 75              | 82.2                               | 10.800          | 15.076         |
| 683-59A                      | 68   | 400              | 10,000         | 290             | 129.8                              | 7.597           | 12.284         |
| <u>CF-3 Grade</u>            |      |                  |                |                 |                                    |                 |                |
| 693-55A                      | 69   | -                | -              | -197            | 194.1                              | 11.249          | 25.251         |
| 693-54A                      | 69   | -                | -              | -50             | 248.4                              | 11.200          | 20.789         |
| 693-53A                      | 69   | -                | -              | 25              | 253.5                              | 9.833           | 17.283         |
| 693-52A                      | 69   | -                | -              | 125             | 373.5                              | 7.636           | 13.612         |
| 693-51A                      | 69   | -                | -              | 290             | 218.1                              | 6.308           | 11.239         |
| 694-51A                      | 69   | -                | -              | -120            | 242.7                              | 10.966          | 23.474         |
| 694-52A                      | 69   | -                | -              | -100            | 258.5                              | 11.219          | 22.800         |
| 694-53A                      | 69   | -                | -              | -10             | 249.9                              | 10.780          | 18.865         |
| 694-54A                      | 69   | -                | -              | 75              | 280.3                              | 8.886           | 14.588         |
| 694-55A                      | 69   | -                | -              | 200             | 246.6                              | 7.187           | 12.323         |
| 69-305A                      | 69   | 320              | 1,000          | 25              | 244.7                              | 9.999           | 17.088         |
| 69-306A                      | 69   | 320              | 1,000          | 25              | 270.7                              | 10.223          | 17.391         |
| 69-307A                      | 69   | 320              | 3,000          | 25              | 271.8                              | 10.175          | 17.469         |
| 69-308A                      | 69   | 320              | 3,000          | 25              | 258.2                              | 10.487          | 17.723         |
| 69-309A                      | 69   | 320              | 10,000         | 25              | 289.4                              | 11.132          | 18.699         |
| 69-310A                      | 69   | 320              | 10,000         | 25              | 231.5                              | 10.741          | 18.015         |
| 69-318A                      | 69   | 350              | 100            | 25              | 261.8                              | 10.516          | 17.498         |
| 692-56A                      | 69   | 350              | 100            | 25              | 235.3                              | 10.145          | 16.688         |
| 69-319A                      | 69   | 350              | 300            | 25              | 248.2                              | 10.175          | 17.186         |
| 692-57A                      | 69   | 350              | 300            | 25              | 234.3                              | 10.194          | 17.879         |
| 69-301A                      | 69   | 350              | 1,000          | 25              | 212.2                              | 10.487          | 17.996         |
| 692-59A                      | 69   | 350              | 1,000          | 25              | 219.2                              | 10.380          | 17.332         |
| 69-320A                      | 69   | 350              | 3,000          | 25              | 229.0                              | 10.477          | 18.230         |
| 692-58A                      | 69   | 350              | 3,000          | 25              | 203.8                              | 10.272          | 17.225         |
| 69-303A                      | 69   | 350              | 10,000         | 25              | 143.4                              | 10.780          | 17.723         |
| 69-311A                      | 69   | 400              | 50             | 25              | 232.2                              | 10.136          | 17.459         |
| 691-56A                      | 69   | 400              | 50             | 25              | 228.2                              | 10.526          | 17.361         |
| 69-314A                      | 69   | 400              | 100            | 25              | 236.5                              | 10.438          | 18.133         |
| 691-59A                      | 69   | 400              | 100            | 25              | 230.6                              | 10.946          | 18.035         |
| 69-312A                      | 69   | 400              | 300            | 25              | 248.9                              | 10.682          | 18.523         |

Table 4. (Contd.)

| Specimen Number <sup>a</sup> | Heat | Aging Temp. (°C) | Aging Time (h) | Test Temp. (°C) | Impact Energy (J/cm <sup>2</sup> ) | Yield Load (kN) | Max. Load (kN) |
|------------------------------|------|------------------|----------------|-----------------|------------------------------------|-----------------|----------------|
| 691-57A                      | 69   | 400              | 300            | 25              | 180.2                              | 10.751          | 17.625         |
| 69-313A                      | 69   | 400              | 1,000          | 25              | 167.8                              | 10.946          | 18.338         |
| 691-58A                      | 69   | 400              | 1,000          | 25              | 129.9                              | 10.839          | 17.273         |
| 69-316A                      | 69   | 400              | 3,000          | 25              | 138.5                              | 10.555          | 17.410         |
| 691-60A                      | 69   | 400              | 3,000          | 25              | 155.3                              | 11.464          | 17.459         |
| 691-54A                      | 69   | 400              | 10,000         | -197            | 39.3                               | 12.831          | 17.498         |
| 692-51A                      | 69   | 400              | 10,000         | -120            | 118.5                              | 11.171          | 19.343         |
| 691-55A                      | 69   | 400              | 10,000         | -50             | 109.9                              | 13.690          | 19.900         |
| 693-60A                      | 69   | 400              | 10,000         | 25              | 103.7                              | 11.571          | 17.635         |
| 694-60A                      | 69   | 400              | 10,000         | 25              | 130.2                              | 11.874          | 17.508         |
| 692-52A                      | 69   | 400              | 10,000         | 75              | 161.9                              | 8.095           | 14.364         |
| 692-53A                      | 69   | 400              | 10,000         | 290             | 129.6                              | 7.528           | 12.635         |
| <u>CF-8M Grade</u>           |      |                  |                |                 |                                    |                 |                |
| 754-57A                      | 75   | -                | -              | -197            | 86.6                               | 18.142          | 24.128         |
| 753-60A                      | 75   | -                | -              | -50             | 198.0                              | 13.631          | 21.003         |
| 753-59A                      | 75   | -                | -              | 25              | 228.3                              | 12.264          | 18.865         |
| 753-58A                      | 75   | -                | -              | 125             | 186.7                              | 10.116          | 15.770         |
| 753-57A                      | 75   | -                | -              | 290             | 201.5                              | 8.134           | 13.524         |
| 754-60A                      | 75   | -                | -              | -197            | 103.8                              | 18.601          | 25.300         |
| 754-58A                      | 75   | -                | -              | -120            | 205.2                              | 16.717          | 25.192         |
| 754-59A                      | 75   | -                | -              | -100            | 174.8                              | 16.453          | 24.987         |
| 754-52A                      | 75   | 320              | 3,000          | 25              | 184.8                              | 12.821          | 18.953         |
| 754-53A                      | 75   | 320              | 3,000          | 25              | 209.2                              | 12.411          | 18.709         |
| 754-54A                      | 75   | 320              | 10,000         | 25              | 185.4                              | 13.153          | 19.832         |
| 754-55A                      | 75   | 320              | 10,000         | 25              | 170                                | 14.041          | 18.738         |
| 752-59A                      | 75   | 350              | 300            | 25              | 234.1                              | 13.133          | 19.822         |
| 752-60A                      | 75   | 350              | 300            | 25              | 244.8                              | 13.084          | 20.007         |
| 753-51A                      | 75   | 350              | 1,000          | 25              | 182.8                              | 13.182          | 19.675         |
| 753-52A                      | 75   | 350              | 1,000          | 25              | 180.6                              | 13.172          | 19.226         |
| 753-53A                      | 75   | 350              | 3,000          | 25              | 115.9                              | 13.426          | 18.416         |
| 753-54A                      | 75   | 350              | 3,000          | 25              | 122.8                              | 13.270          | 19.343         |
| 753-55A                      | 75   | 350              | 10,000         | 25              | 82.4                               | 14.705          | 19.500         |
| 754-51A                      | 75   | 350              | 10,000         | 25              | 33.7                               | 15.467          | 15.789         |
| 751-56A                      | 75   | 400              | 100            | 25              | 227.0                              | 13.250          | 20.525         |
| 751-57A                      | 75   | 400              | 100            | 25              | 223.9                              | 12.801          | 19.666         |
| 751-58A                      | 75   | 400              | 300            | 25              | 208.0                              | 13.573          | 20.955         |
| 751-59A                      | 75   | 400              | 300            | 25              | 189.8                              | 13.553          | 20.369         |
| 751-60A                      | 75   | 400              | 1,000          | 25              | 86.2                               | 13.553          | 17.683         |
| 752-56A                      | 75   | 400              | 1,000          | 25              | 68.3                               | 13.700          | 16.502         |
| 752-57A                      | 75   | 400              | 3,000          | 25              | 57.3                               | 13.827          | 16.756         |
| 752-58A                      | 75   | 400              | 3,000          | 25              | 70.0                               | 13.963          | 17.098         |
| 75-301A                      | 75   | 400              | 10,000         | -197            | 9.1                                | 15.330          | 15.330         |
| 75-302A                      | 75   | 400              | 10,000         | -50             | 30.7                               | 16.277          | 16.277         |
| 75-365A                      | 75   | 400              | 10,000         | 25              | 43.4                               | 14.871          | 16.531         |
| 75-366A                      | 75   | 400              | 10,000         | 25              | 37.7                               | 15.330          | 16.980         |
| 75-303A                      | 75   | 400              | 10,000         | 75              | 62.4                               | 13.211          | 16.404         |
| 75-304A                      | 75   | 400              | 10,000         | 290             | 95.4                               | 9.970           | 14.334         |

<sup>a</sup> All specimens aged 10,000 h at 400°C and annealed for 1 h at 550°C and water quenched prior to aging.

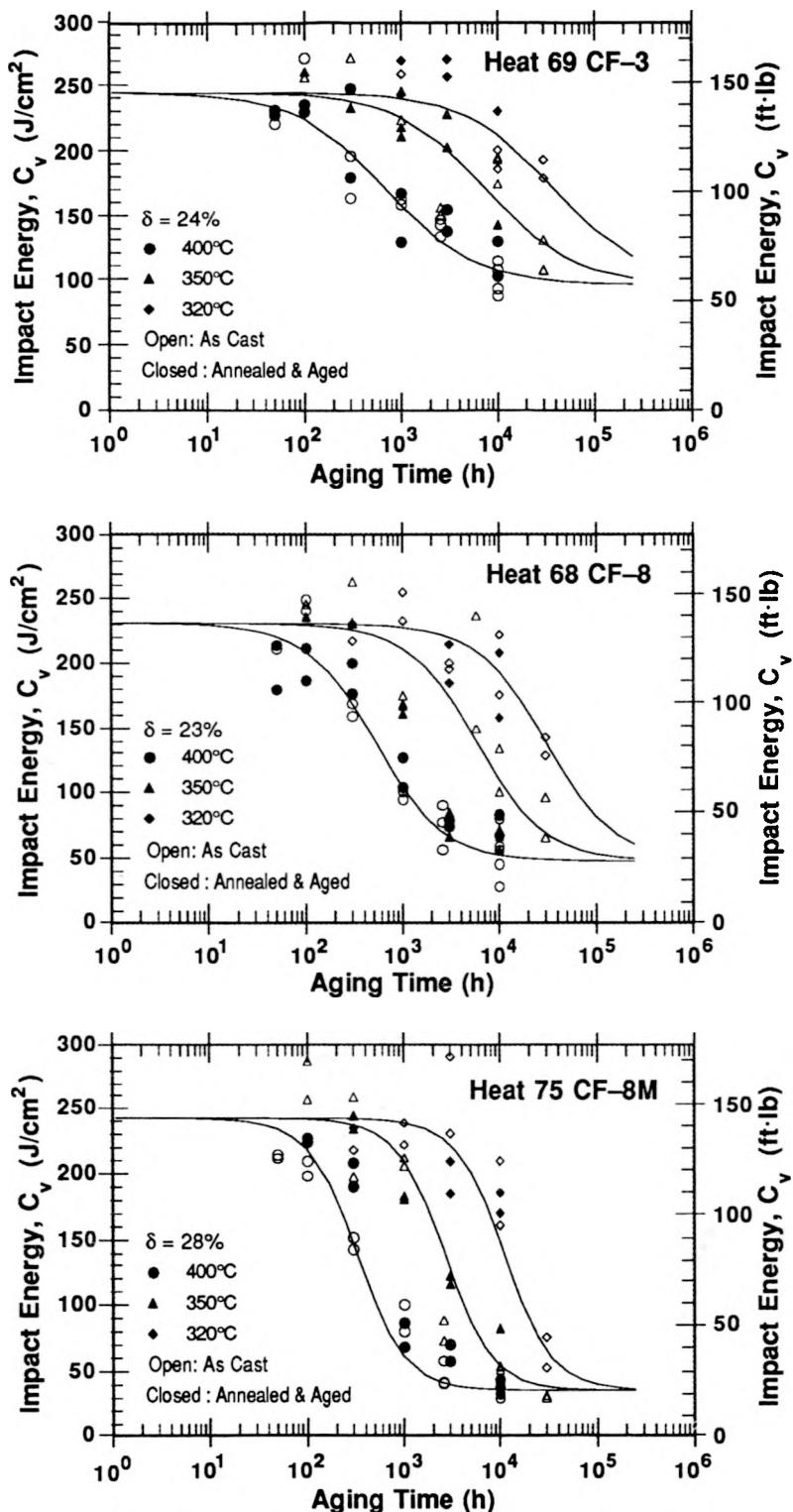


Figure 18. Effect of thermal aging on room-temperature impact energy of unaged and recovery-annealed CF-3, CF-8, and CF-8M steel. The solid lines represent the aging behavior of the as-cast materials.

are slightly faster than the kinetics of embrittlement of the as-cast materials. However, there is significant overlap in the 95% confidence limits between the values obtained for as-cast and recovery-annealed materials. The difference in the kinetics of Heats 68 and 75 is primarily due to the aging behavior at 400°C. The decrease in impact energy at 320°C is comparable for as-cast and recovery-annealed materials.

The results indicate that baseline mechanical properties and possibly the kinetics of embrittlement of cast stainless steels can be determined from the recovery-annealed material. This may be very useful for evaluating degradation of mechanical properties of service-aged components, where the baseline mechanical properties or archive material are generally not available.

## 4 Conclusions

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Charpy-impact data are presented for several experimental and commercial heats of cast stainless steel that were aged up to 30,000 h at temperatures of 290–450°C. The results indicate that, for a specific cast stainless steel, the extent of embrittlement, i.e., the minimum room-temperature impact energy that would be achieved after long-term aging, depends on the chemical composition and ferrite morphology of the steel. A correlation is presented for estimating the extent of embrittlement from known material information.

The results indicate that the extent and rate of embrittlement depend on material parameters, i.e., ferrite morphology and chemical composition of the steel. Ferrite morphology strongly affects the extent of embrittlement, whereas material composition influences the kinetics of embrittlement. Small changes in the constituent elements of the cast material can cause the kinetics of embrittlement to vary significantly.

Annealing studies indicate that the degradation in mechanical properties due to thermal embrittlement can be reversed by annealing the embrittled material for 1 h at 550°C and then water quenching. The kinetics of embrittlement can also be obtained from aging studies on the reembrittlement of recovery-annealed material. This procedure may be useful for establishing the baseline mechanical properties and kinetics of embrittlement of service-aged components.

## Acknowledgments

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This progress report summarizes work performed by Argonne National Laboratory on long-term embrittlement of cast duplex stainless steels in LWR systems during the six months from October 1988 to March 1989. Charpy-impact data are presented for several heats of cast stainless steel aged at temperatures between 320 and 450°C for times up to 30,000 h. Thermal aging decreases impact energy and shifts transition curves to higher temperatures. A saturation effect is observed for room-temperature impact energy and upper-shelf energy. Charpy data are analyzed to obtain the activation energy of the kinetics of embrittlement. The results suggest that the activation energy of embrittlement is not constant in the temperature range of 290–400°C, but increases as temperature decreases. A correlation is presented for estimating the extent of embrittlement of cast stainless steels from known material parameters. The degradation in mechanical properties can be reversed by annealing the embrittled material for 1 h at 550°C and then water quenching.

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