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RADIATION-INDUCED PHASE DEVELOPMENT IN AISI 316

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RADIATION-INDUCED PHASE DEVELOPMENT IN AISI 316*

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The evolution of phases during fast reactor irradiation of AISI 316 has been studied in three different experiments to fluences as high as 1.4×10^{23} n/cm² ($E > 0.1$ MeV). When added to insight gained from previous experiments, a comprehensive picture emerges of the dependency of phase evolution in this alloy on both the preirradiation thermomechanical treatment and the in-reactor temperature history. The identity and volume fraction of precipitates at a given temperature change not only at different points within a grain but also between nominally identical heats subjected to the same thermomechanical treatment.

One feature common to these steels is the selective concentration of the elements nickel and silicon into all precipitates. The precipitates formed during irradiation can be classified into four types. These are (1) precipitates such as $M_{23}C_6$ and Laves phase which are progressively changed in composition by an infiltration-exchange process, (2) precipitates such as η -silicide which are naturally rich in nickel and silicon, (3) precipitates such as γ' and G-phase (Mn-Ni silicide) which form only under irradiation, and (4) precipitates such as M_6C which appear to result from the transformation of $M_{23}C_6$ particles due to composition changes induced by irradiation.

It is concluded that irradiation causes a major perturbation in the microchemistry and microstructure of AISI 316, and that these perturbations are sensitive to preirradiation composition and thermomechanical treatments.

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Introduction

The identity and kinetics of the precipitate phases formed at elevated temperature in unirradiated AISI 316 stainless steel have been documented by a number of investigators (1-3). However, irradiation has been shown to modify both the stability of the austenite matrix and the precipitation reactions in this steel (4-11). For instance, the phases γ' (Ni_3Si) (4,9) and G (a nickel silicide) (10,11) are formed in 316 stainless steels only during irradiation and would not otherwise be produced. The formation of these irradiation-induced precipitates has also been shown to affect the matrix composition by depleting it of certain solute elements. A similar irradiation-induced concentration of these solutes in the thermally stable phases has also been observed (6). The development of these irradiation-induced and irradiation-altered phases is also expected to affect many properties of the material such as the mechanical properties (12), the creep and swelling behavior (5,13), and possibly the corrosion resistance. This paper reports the nature of various phases observed in AISI 316 during both isothermal and nonisothermal irradiation in the EBR-II fast reactor.

Experimental Procedure

As indicated in Table I, the evolution of precipitate phases in AISI 316 has been studied using specimens from three different experiments. Experiments RS-1 and AA-XI involved isothermal irradiation and the YY07-F experiment was an experiment wherein temperatures were changed during the irradiation. Specimens employed in RS-1 and YY07-F were thin-walled fuel clad tubing in the 20% cold-worked condition, and specimens in AA-XI were small microscopy disks in the solution annealed condition. The steel used in these experiments was derived from four different vendor-produced heats designated DD, CN-13, N-lot and R-lot. The heats DD and CN-13 are vacuum-melted steels made from high purity ingredients and are typical of those employed in the first core of the Fast Flux Test Facility. N-lot is a reference heat employed in the U.S. breeder reactor program. Both N-lot and R-lot are vacuum-melted

Table I. Irradiation Experiments

| Experiment | Irradiation Temperature (°C) | Neutron Fluence (10^{22} n/cm ² , E >0.1 MeV) | Material |
|------------|---|--|---|
| RS-1 | Isothermal irradiation at 467, 500, 533, 567, 600 and 650°C | 14 | 20% Cold Worked CN-13 N-Lot R-Lot |
| AA-XI | Isothermal irradiation at 450, 600 and 650°C | 7 | Solution Annealed CN-13 N-Lot |
| YY07-F | Original irradiation in MV-III, 533, 600 and 650°C | 4.5 | 20% Cold Worked CN-13 N-Lot |
| | Final irradiation at 625°C | 10.0 Total | DD-Lot |

steels containing small but unidentified amounts of scrap metals. These latter two heats are produced by processes which differ from that of the DD and CN-13 heats, having recrystallization anneals of 50 seconds at 1050°C instead of approximately 3 minutes at 1010°C. As indicated in Table II, there are only minor differences in chemical composition among the four heats.

Table II. Composition of Various Heats of 20% Cold Worked
AISI 316 Stainless Steel

| Lot No. | C | Mn | Si | P | S | Cr | Ni |
|---------|-------|------|------|-------|-------|-------|-------|
| N-Lot | 0.056 | 1.64 | 0.46 | 0.013 | 0.006 | 16.52 | 13.66 |
| R-Lot | 0.04 | 1.36 | 0.52 | 0.011 | 0.009 | 17.76 | 13.27 |
| CN-13 | 0.053 | 1.63 | 0.50 | 0.002 | 0.003 | 17.26 | 13.72 |
| DD-Lot | 0.055 | 1.60 | 0.54 | 0.005 | 0.006 | 17.79 | 13.70 |

| Lot No. | Mo | Cu | N | Co | B | Other |
|---------|------|-------|-------|-------|---------|---|
| N-Lot | 2.41 | 0.08 | 0.006 | 0.05 | 0.0008 | |
| R-Lot | 2.53 | 0.08 | 0.012 | 0.032 | 0.001 | |
| CN-13 | 2.25 | <0.01 | 0.004 | 0.01 | <0.0005 | 0.01 V <0.005 Al <0.015 Ta <0.005 As |
| DD-Lot | 2.24 | <0.05 | 0.018 | 0.018 | <0.001 | <0.01 Nb |

The microstructural and microchemical analyses of these specimens were performed *in situ* in a JEOL 200B scanning transmission electron microscope with the aid of energy dispersive X-ray (EDX) analysis. Precipitate phases were identified not only by their diffraction patterns but also by their chemical composition obtained from EDX analysis. The techniques employed have been described elsewhere (14).

Results: Phase Identification

The precipitate phases identified thus far to form in irradiated AISI 316 are $M_{23}C_6$, M_6C , γ' , η -silicide, G, and Laves. With the exception of M_6C , γ' and Laves, the other three phases form throughout the temperature range examined, 450 to 650°C. Gamma prime was detected only at irradiation below approximately 560°C, while M_6C and Laves were detected only above approximately 560°C. The relative amount of these different phases formed during irradiation, however, is sensitive to the chemical composition, irradiation history and preirradiation thermomechanical treatments of each material. The balance of precipitates was frequently found to shift within a single grain.

The phases $M_{23}C_6$, M_6C , η -silicide and G-phase require careful attention in order to distinguish between them. The $M_{23}C_6$ and G-phase possess an fcc structure (space group Fm3m) with lattice parameters of 1.077 and 1.13 nm, respectively. However, $M_{23}C_6$ exhibits a strong parallel relationship with the matrix while G-phase forms primarily in a cube-on-twin relationship to the matrix as well as weak habit relationships of $(110)_G \parallel (111)_\gamma$ and $(110)_G \parallel (100)_\gamma$. η -silicide and M_6C are diamond cube in structure (space

group Fd3M) with a lattice parameter of 1.077 nm. Eta-phase exhibits the same relationship to the matrix as does G-phase while M_6C possesses a cube-on-cube relationship. The latter phase forms only at higher irradiation temperatures, however. Being careful to include double-diffraction effects, M_6C and η -silicide can be identified by inspection of diffraction patterns utilizing low index orientations wherein certain reflections are systematically absent. Typical selected area diffraction patterns from $M_{23}C_6$ and M_6C , G and η -silicide are presented in Figures 1, 2 and 3, respectively.

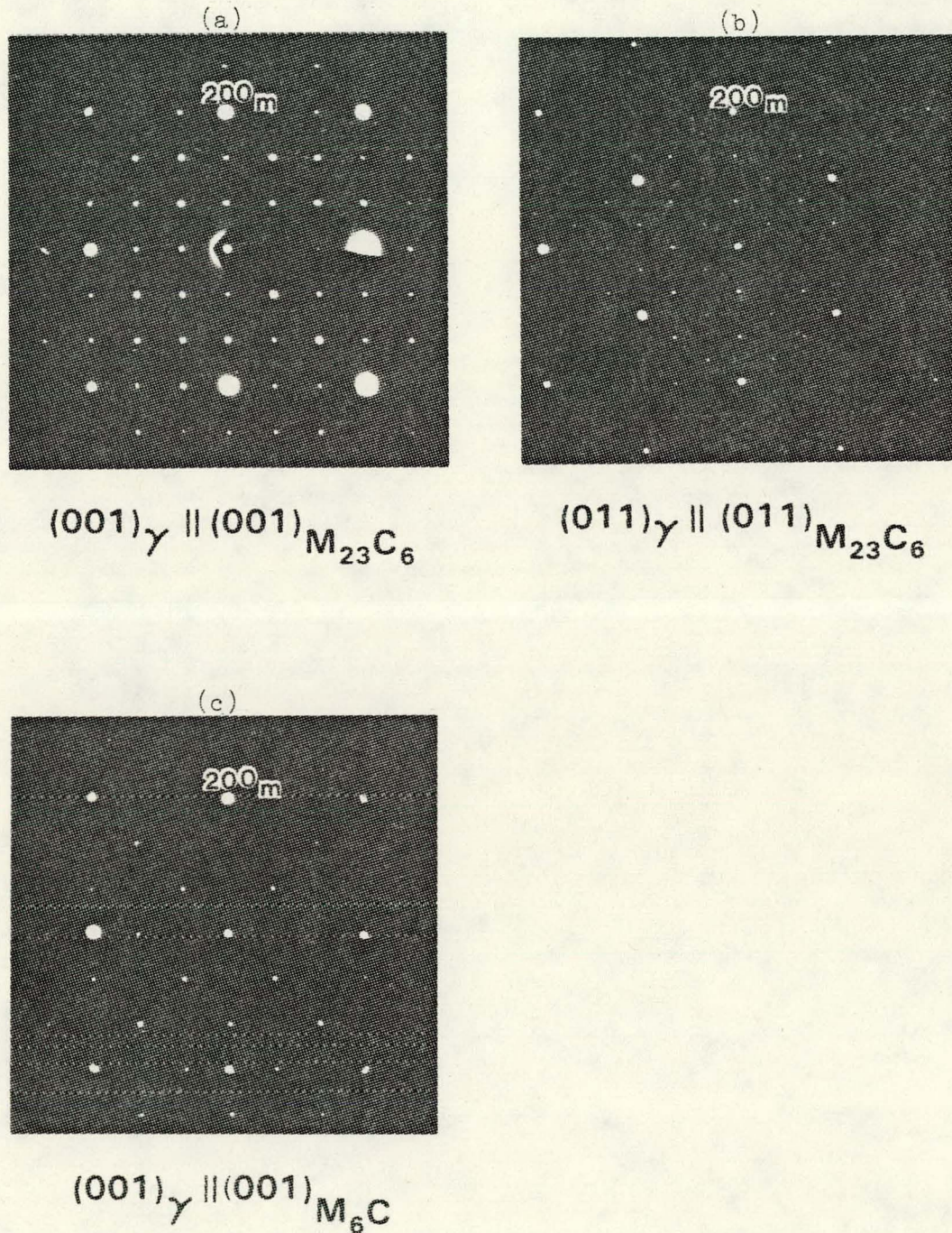


Fig. 1 - Selected area diffraction patterns from $M_{23}C_6$ and M_6C . (a) $(001)_{\gamma} \parallel (001)_{M_{23}C_6}$, $[002]_{\gamma} \parallel [002]_{M_{23}C_6}$, (b) $(011)_{\gamma} \parallel (011)_{M_{23}C_6}$, $[002]_{\gamma} \parallel [002]_{M_{23}C_6}$, (c) $(001)_{\gamma} \parallel (001)_{M_6C}$, $[002]_{\gamma} \parallel [004]_{M_6C}$.

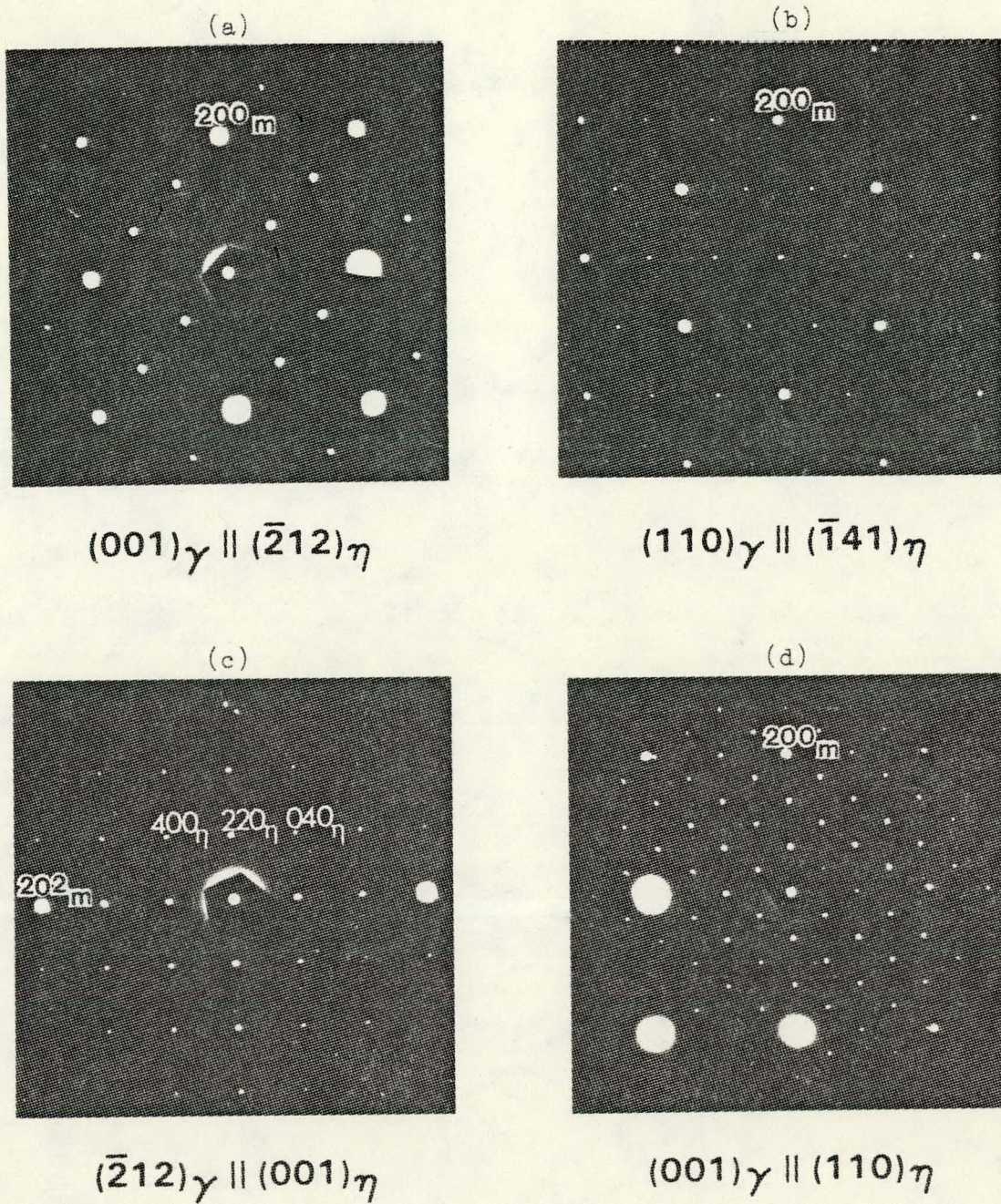


Fig. 2 - Selected area diffraction patterns from η -silicide particles which have mostly cube-on-twin habit relationships to the matrix as:

(a) $(001)_\gamma \parallel (\bar{2}12)_\eta, [220]_\gamma \parallel [202]_\eta.$

(b) $(110)_\gamma \parallel (14\bar{1})_\eta, [202]_\gamma \parallel [220]_\eta.$

(c) $(\bar{2}12)_\gamma \parallel (001)_\eta, [202]_\gamma \parallel [220]_\eta.$

Habit relationship of $(001)_\gamma \parallel (011)_\eta, [002]_\gamma \parallel [002]_\eta$ has also been observed as in (d).

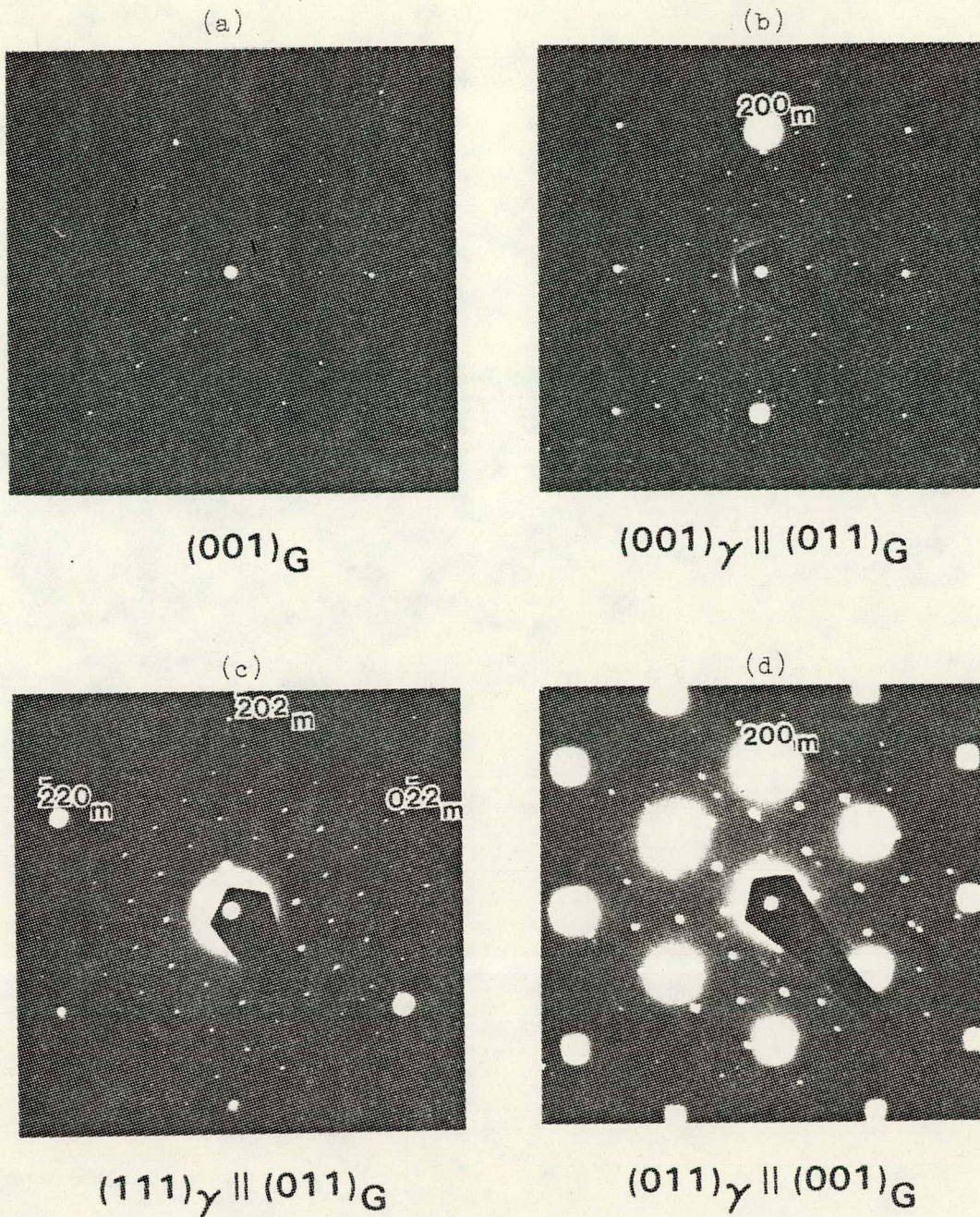


Fig. 3 - Selected diffraction patterns from G-phase particles with habit relationships to the matrix other than cube-on-twin.

- (a) $(001)_G$ pattern; the particle does not have a particular habit relationship with respect to the matrix.
- (b) $(001)_\gamma || (011)_G$.
- (c) $(111)_\gamma || (011)_G$.
- (d) $(011)_\gamma || (001)_G$.

Results of the RS-1 Experiment

All heats except the DD heat were irradiated together in this experiment in the 20% cold-worked condition in sodium-filled capsules. Although nominally similar in composition, each heat of steel developed not only different amounts of phases for a given set of irradiation conditions, but also different amounts

of swelling (15). Table III contains a compilation of the various precipitate phases observed in each heat of steel as a function of temperature. Each phase is listed in order of decreasing abundance. In any one heat there appears to exist a variety of competing phases with the predominant species shifting from γ' and $M_{23}C_6$ at lower temperatures to η -silicide, G and Laves at higher temperatures. The level of γ' in the three heats is different not only in amount but also in its observed range in temperature. The R-lot heat has the least amount of γ' of the three heats and the smallest temperature regime of stability at this fluence. The CN-13 heat has the largest amount of G-phase of the three heats, but no G-phase is detected at any temperature in the R-lot heat. The formation rate of both intermetallic phases and γ' in N-lot is known to be rather slow relative to that of CN-13 and DD heat (4,16,17).

Table III. Precipitate Phases* Observed in 20% Cold Worked AISI 316 Irradiated in the RS-1 Experiment to 14×10^{22} n/cm² (E > 0.1 MeV)

| Irradiation Temperature (°C) | CN-13 | N-Lot | R-Lot |
|------------------------------|---|---|--|
| 467 | γ' $M_{23}C_6$ G η | γ' $M_{23}C_6$ η G | $M_{23}C_6$ η |
| 500 | γ' $M_{23}C_6$ G η | γ' $M_{23}C_6$ η G | $M_{23}C_6$ γ' η |
| 533 | $M_{23}C_6$ γ' G η | γ' $M_{23}C_6 + M_6C^{**}$ η G | γ' $M_{23}C_6$ η |
| 567 | G η $M_{23}C_6$ | $M_{23}C_6 + M_6C^{**}$ γ' η G | $M_6C + M_{23}C_6^{**}$ η |
| 600 | $M_{23}C_6 + M_6C^{**}$ Laves η G | $M_6C + M_{23}C_6^{**}$ Laves η G | $M_6C + M_{23}C_6^{**}$ Laves η |
| 650 | Laves $M_6C + M_{23}C_6^{**}$ η G | $M_6C + M_{23}C_6^{**}$ Laves η G | Laves Small amounts of $M_{23}C_6 + M_6C^{**}$ and η |

* Phases are listed in order of predominance.

** Both phases appear simultaneously and usually in association with each other.

The compositions of the various precipitates found in these specimens are listed in Table IV. All phases are unexpectedly rich in nickel and silicon. Note that η -silicide generally contains higher levels of nickel and silicon

Table IV. Average Compositions of Precipitate Observed in Neutron Irradiated 20% CW AISI 316 Stainless Steels

| Steel | Precipitate Particle | Wt. Percent | | | | | |
|-------|--------------------------------|-------------|------|------|------|------|-----|
| | | Si | Cr | Fe | Ni | Mo | Mn |
| CN-13 | M ₂₃ C ₆ | 0.4 | 20.7 | 64.3 | 10.7 | 3.8 | - |
| | η-silicide | 3.0 | 24.4 | 51.6 | 16.9 | 4.0 | - |
| | G-Phase | 15.0 | 4.9 | 9.5 | 59.9 | 5.0 | 5.7 |
| | Laves | 3.4 | 15.8 | 44.2 | 21.8 | 14.4 | - |
| N-Lot | M ₂₃ C ₆ | 1.0 | 17.8 | 65.6 | 10.4 | 4.1 | - |
| | η-silicide | 3.5 | 19.0 | 51.9 | 19.6 | 5.3 | - |
| | G-phase | 16.6 | 4.3 | 8.7 | 62.3 | 2.4 | 5.6 |
| | Laves | 4.2 | 16.4 | 37.6 | 27.7 | 13.6 | - |
| R-Lot | M ₂₃ C ₆ | 0.4 | 22.2 | 63.2 | 11.2 | 2.9 | - |
| | η-silicide | 2.4 | 17.4 | 61.6 | 15.4 | 3.0 | - |
| | Laves | 1.4 | 13.8 | 41.7 | 21.0 | 21.9 | - |

than found in M₂₃C₆. Both η-silicide and the M₆C observed at higher temperatures have a diamond cubic structure, and both have been cataloged as η-carbides by Goldschmidt (18) and Stadelmaier (19). The M₆C particles are frequently found to be associated with M₂₃C₆ particles as illustrated in Figure 4. The chemical composition of M₆C could not be accurately determined since the X-ray spectra from associated M₆C and M₂₃C₆ were always superimposed.

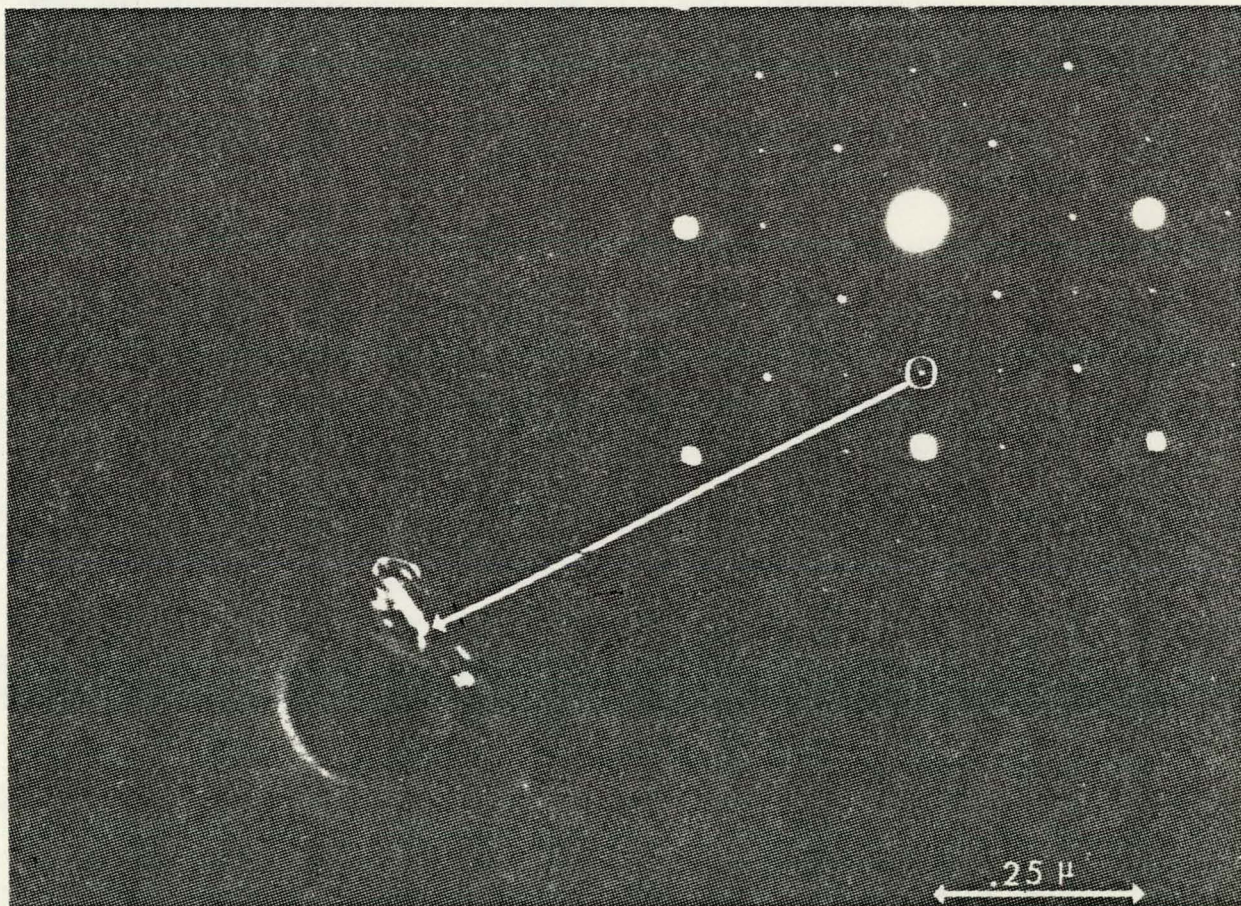


Fig. 4 - Close association between M₂₃C₆ and M₆C particles. Precipitate particles are in dark-field images from 004 reflection.

G-phase was found to be enriched in Mn, Ni, and Si. G-phase is a silicide with an ideal composition of $Ti_6Ni_{17}Si_7$. Figure 5 shows a typical example of two precipitate phases (G and γ') coexisting in the same grain.

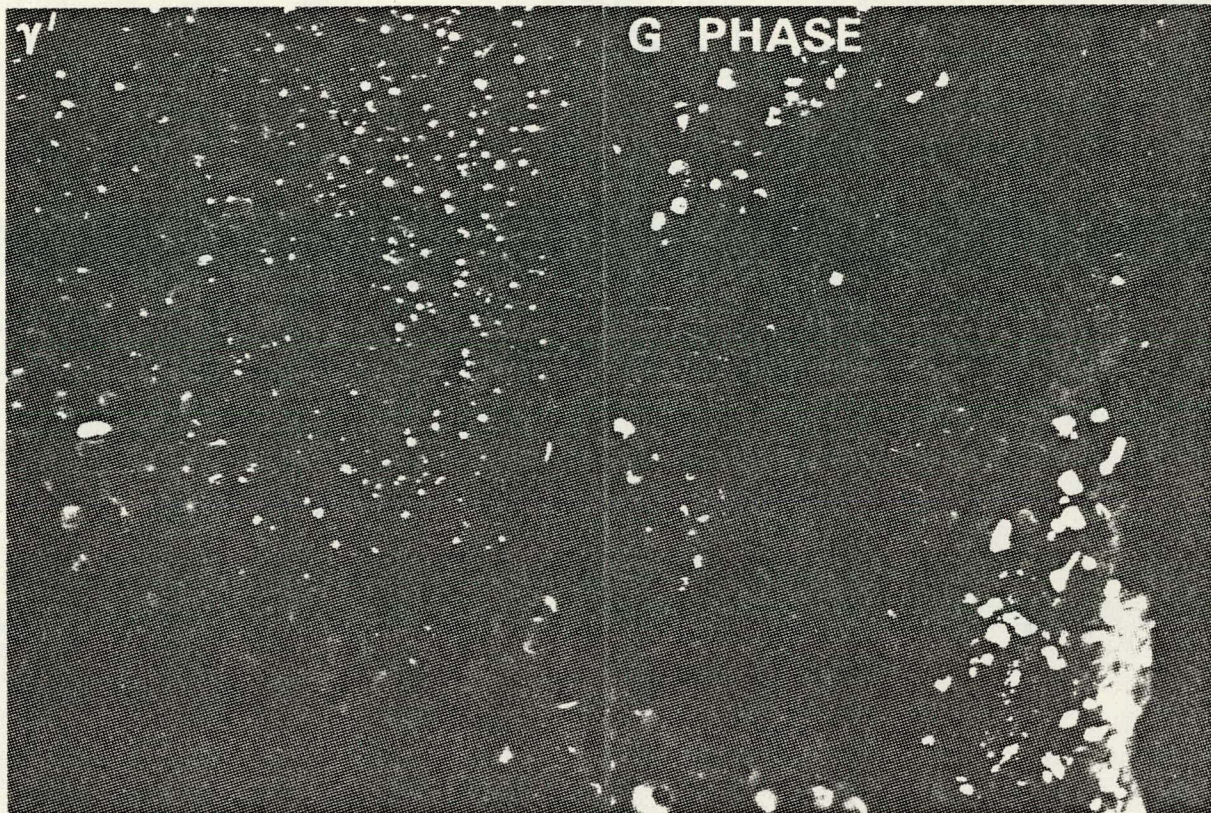


Fig. 5 - Gamma prime and G-phase coexisting in the same grain of a CN-13 sample irradiated at 500°C to 14×10^{22} n/cm² (E > 0.1 MeV).

Results of the AA-XI Experiment

Whereas the cold-worked material described in the previous section was from commercially drawn tubing, the AA-XI specimens were punched as disks from cold-rolled bars and then solution annealed at 1100°C for 15 minutes. This is a substantially different annealing treatment than the 50 seconds at 1050°C treatment experienced by the tubing prior to or between cold-working passes. Cold-working has been shown to substantially alter the phase development of the N-lot heat of this steel (5).

Three specimens from this experiment have been examined. In the first (N-lot, 650°C, 7×10^{22} n/cm²), $M_{23}C_6$ was found to be the predominant phase with the following composition (in wt. %): Cr: 35; Fe: 8.5; Ni: 34; Mo: 15.6; and Si: 6.5. Both η -silicide and M_6C were found at very low densities and there were no Laves or G-phase in this specimen as was found in the cold-worked steel irradiated in the RS-1 experiment.

The second specimen was also an N-lot disk but was irradiated at 450°C to 7×10^{22} n/cm². It contained only γ' in the matrix, with some $M_{23}C_6$ at the grain boundaries. It did not contain the balance of γ' , $M_{23}C_6$, η -silicide and G-phase found in the matrix of the cold-worked specimen irradiated at a comparable temperature in the RS-1 experiment.

The third specimen was derived from the CN-13 heat irradiated to 7×10^{22} n/cm² at 600°C and contained no Laves precipitates, a low density of $M_{23}C_6$ in

the matrix and a moderate density of G-phase and η -silicide, with $M_{23}C_6$ and G-phase usually associated with voids. The $M_{23}C_6$ and η -silicide precipitates have similar compositions (in wt. %): Cr: 37.5; Fe: 8; Ni: 34.5; Mo: 11.6; and Si: 8. Note that once again the precipitate balance is altered from that found in the cold-worked specimen irradiated in the RS-1 experiment at the same temperature. Note also that the $M_{23}C_6$ in this annealed steel contained higher levels of Cr, Ni, Mo and Si than that found in the same heat of 20% CW 316 irradiated in RS-1, even though the fluence attained by the annealed steel was about one half of that reached by the cold-worked steel.

Results of the YY07-F Temperature Change Experiment

The YY07 experiment involved the irradiation of clad tubing specimens (N, CN-13 and DD) to higher fluence at a temperature of 625°C after prior irradiation in the RS-1 experiment to 4 to 5 x 10²² n/cm² (E > 0.1 MeV) at temperatures of either 533, 600 or 650°C. All nine specimens (three heats at each starting temperature) had precipitate microstructures typical of that produced in isothermal experiments at about 625°C. The precipitates were predominantly Laves along with $M_{23}C_6$, η -silicide, and G-phase in lower densities. The chemical compositions of the various precipitates agree with the compositions found in the isothermal irradiation experiment, RS-1.

It is important to note that no γ' phase was found in any of these specimens, including those originally irradiated at 533°C where γ' is known to form in the CN-13 and DD heats at 4 to 5 x 10²² n/cm². The γ' formed in this heat at 533°C has obviously dissolved during irradiation at 625°C. This is in agreement with the previous findings that γ' in this steel is stable only at lower temperatures and dissolves at higher temperature (4,5)

Discussion

As shown in this and other studies (5,16) the phase development of a given heat of AISI 316 is quite sensitive to the preirradiation thermo-mechanical treatment and the temperature history during irradiation. The balance of precipitates which evolve is also quite sensitive to minor compositional differences, and irradiation temperature and also appears to evolve with fluence. Some precipitates show stability at low temperatures but once formed can dissolve if the irradiation temperature is increased. It has been concluded from this and other efforts (4-6,20) that different 316 heats in all starting conditions are progressing toward a state wherein the nickel concentration of the matrix is reduced to a level determined primarily by the silicon content (5,6). Although the phase evolution of AISI 316 has been shown to be highly variable, the matrix composition is proceeding toward a level that is relatively independent of the route of precipitation and segregation. The elements nickel and silicon are found to concentrate into all precipitates by one of two mechanisms. $M_{23}C_6$ and Laves normally form with very low levels of nickel and silicon and become progressively enriched in these elements during irradiation. Gamma prime, η -silicide and G-phase appear to be naturally rich in nickel and silicon and require irradiation for their formation and continued stability.

It appears that the precipitates induced in irradiated AISI 316 can be grouped into the following four classes:

Class I: Thermally Stable Phases

These are the precipitates normally found in aged AISI 316, $M_{23}C_6$ and Laves (21-23). The intermetallics sigma and chi are also in this group but have not yet formed at the temperatures and times experienced by the specimens examined in these studies. The formation and growth of these phases proceeds by a mechanism of absorbing vacancies from the matrix (21). They form with very low levels of nickel and silicon during thermal aging. The process by which silicon and nickel are enriched in these precipitates has been designated as "infiltration-exchange" (6,14,20).

Class II: Irradiation Assisted Phases

In alloys with higher silicon levels, the η -silicide forms naturally without irradiation, although radiation may assist its rate of formation. Maziasz (24) first identified the η -silicide in AISI 316, noting that it formed not only in-reactor (both EBR-II and HFIR), but also in specimens aged out-of-reactor at temperatures ranging from 600 to 700°C for times varying from 1,000 to 10,000 hours. This phase is naturally rich in chromium, nickel and silicon and the composition does not change in-reactor. The DO heat of AISI 316 examined by Maziasz is richer in silicon, 0.8 wt. % (substantially higher than the 0.5 wt. % present in the heats of this study). In an unpublished study by Yang, the prototypic 20% CW heats CN-13 and N-lot, after 3,000 and 15,000 hours in the temperature range of 590 to 760°C, did not produce η -silicide. Since η -silicide was observed in the irradiated and not the aged unirradiated conditions, radiation probably at least assists its growth.*

Class III: Irradiation-Induced Phases

G-phase and γ' precipitates are naturally rich in nickel and silicon and form in AISI 316 only during irradiation, although the G-phase forms over a wider range of temperatures than does γ' . Once formed at low temperatures, the γ' tends to dissolve when the irradiation temperature is increased. If the silicon content of the alloy is increased the stability of the γ' phase is extended to higher temperature, i.e., 620°C for 1.5 wt. % silicon (27). If the temperature is maintained but the irradiation ceases, both phases start to dissolve, indicating that both phases require irradiation for continued stability. Additional specimens of the irradiated CN-13 material shown in Figure 5 were annealed at 500°C for 1,000 hours and both phases had completely dissolved. The dissolution is rather sluggish since a large fraction of the original precipitates were still present in another specimen annealed only 500 hours. These findings are in agreement with those of an earlier study (5).

Class IV: Transformed Phases

The M_6C may be the result of a transformation at higher irradiation temperatures of $M_{23}C_6$ possibly as a result of increased nickel and silicon content resulting from the infiltration-exchange process. The strongest evidence

* The η -silicide has also been observed in other high silicon austenitic steels by Hughes (25) and Tither and Clark (26). Hughes first observed a diamond cubic phase together with $M_{23}C_6$ in aged 12Cr-4Ni-3Mn-3Si-1V-0.03C steel and referred to as "H" phase. This phase was also observed in aged 18Cr-15Ni-4.5Si-1Mn-0.03C steel by Tither and Clark, but they misidentified the phase as a fcc crystal structure, although the X-ray diffraction patterns showed it to be diamond cubic.

for this supposition is the frequent association of $M_{23}C_6$ - M_6C bi-crystals. However, M_6C has also been observed in aged 316L by Weiss and Stickler in the absence of irradiation (1). Inoue and Masumoto (28) reported that the transformation from $M_{23}C_6$ to M_6C has been observed in aged Fe-Cr-Mo and Fe-Cr-W steels containing high carbon.

Conclusion

During irradiation at temperatures between 460 and 650°C, the alloy AISI 316 decomposes into an austenite matrix of altered composition and some mixture of six possible precipitate phases. These phases are γ' , G, η -silicide, $M_{23}C_6$, M_6C and Laves. The balance of phases developed is exceptionally sensitive to a large number of material and environmental variables and frequently varies within a single grain. All of these phases are found to be either naturally rich in nickel and silicon or to become progressively enriched in these elements as the irradiation proceeds. The precipitates can be considered to be classified as thermally stable but modified, irradiation-enhanced, irradiation-induced and irradiation-transformed.

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