ANOMALOUSLY LARGE DEFORMATION OF 12Cr18Ni10Ti AUSTENITIC STEEL IRRADIATED TO 55 DPA AT 310ºC IN THE BN-350 REACTOR - M. N. Gusev, O. P. Maksimkin , I. S. Osipov (Institute of Nuclear Physics, Kazakhstan) and F. A Garner (Pacific Northwest National Laboratory)*

OBJECTIVE

The objective of this effort is to determine the response to irradiation of austenitic steels at high neutron exposures.

SUMMARY

Whereas most previous irradiation studies conducted at lower neutron exposures in the range 100-400ºC have consistently produced strengthening and strongly reduced ductility in stainless steels it now appears possible that higher exposures may lead to a reversal in ductility loss for some steels. A new radiation-induced phenomenon has been observed in 12Cr18Ni10Ti stainless steel irradiated to 55 dpa. It involves “a moving wave of plastic deformation” at 20ºC that produces “anomalously” high values of engineering ductility, especially when compared to deformation occurring at lower neutron exposures. Using the technique of digital optical extensometry the “true stress σ –true strain ε” curves were obtained. It was shown that a moving wave of plastic deformation occurs as a result of an increase in the intensity of strain hardening, $d\sigma/d\varepsilon(\varepsilon)$. The increase in strain hardening is thought to arise from an irradiation-induced increase in the propensity of the $\gamma\rightarrow\alpha$ martensitic transformation.

PROGRESS AND STATUS

Introduction

It is generally accepted that irradiation of stainless steels at temperatures of 100-400ºC leads to a rapid increase in strength and a concurrent reduction in both uniform and total elongation during deformation, a behavior that is clearly seen in “engineering” stress-strain curves and that is almost always associated with early flow localization leading to necking.

Using a technique called “digital marker extensometry”, however, we have shown recently that the stress-strain deformation characteristics (true stress-true deformation) continue unchanged in the necking region even though the remainder of the specimen no longer participates in the deformation process [1, 2]. Figures 1 and 2 demonstrate this behavior for stainless steels irradiated in two different reactors. These results signal that a distinction should be made between “true embrittlement” involving suppression of material capability for plastic deformation and “quasi-embrittlement” involving a reduction of uniform and total deformation as a result of development of a macroscopic neck.

Another well-accepted perception is that continued neutron exposure quickly leads to a saturation in mechanical properties that remains unchanged until significant void swelling is attained [3-7]. It now appears that this perception must be at least partially modified for relatively low irradiation temperature and very high fluence exposure, especially for steels prone to an austenite to martensite instability. In this paper we demonstrate that the trend toward reduced elongation with increasing exposure can be reversed at very high dose.

* Pacific Northwest National Laboratory (PNNL) is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RLO-1830.

Experimental Procedure

A hexagonal wrapper constructed from 12Cr18Ni10Ti steel was removed from a spent fuel assembly designated CC-19 after irradiation in the reflector region of the BN 350 fast reactor core. The wrapper walls were 2 mm thick with face-to-face distance of 96 mm. Prior to irradiation the wrapper was formed with a final cold deformation of 15 to 20%, followed by annealing at 800°C for an hour. During irradiation the wrapper reached a maximum dose and temperature of 55 dpa and 310°C. The inlet temperature of 280ºC defines the lowest temperature of the wrapper.

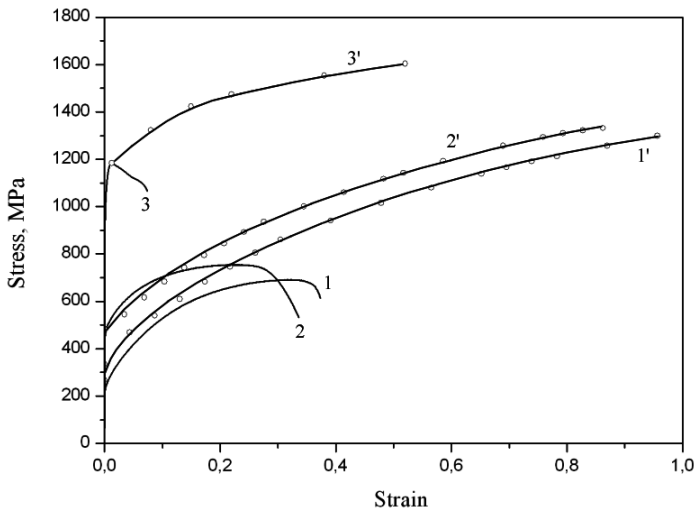

Figure 1. Engineering curves (1, 2, 3) and "$\sigma_i - \varepsilon_i$" relationships (1', 2', 3') for non-irradiated (1) and irradiated (2, 3) stainless steels tested at 20ºC. 2 – 12Cr18Ni10Ti (WWR-K thermal reactor, 1.4×10^{19} n/cm^2 and 80ºC), 3 – 08Cr16Ni11Mo3 (BN-350 fast reactor, 15.6 dpa and 340ºC).

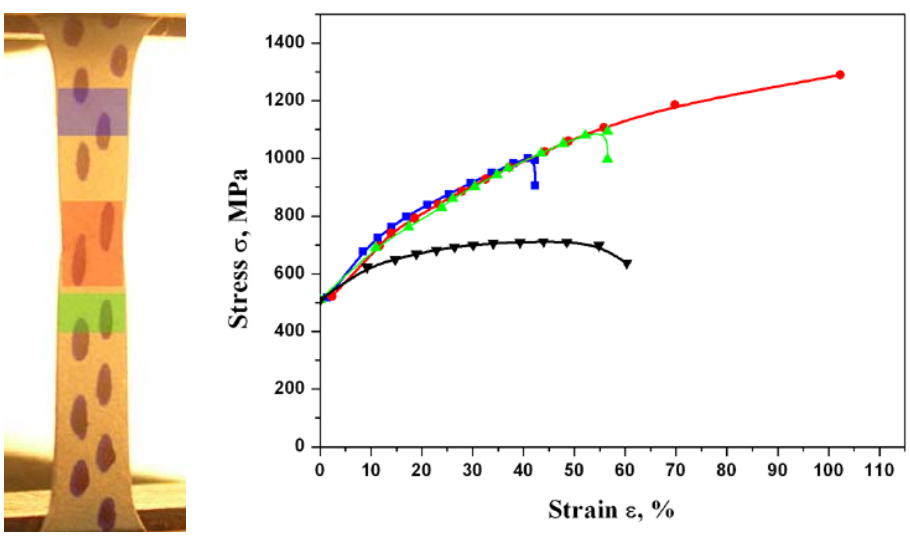

Figure 2. 12Cr18Ni10Ti irradiated to 1.4×10^{19} n/cm^2 (E>0.1) in the WWR-K reactor at 80ºC and tested at 20ºC. Comparison of engineering (black) and true strain curves (designated by colors) for three areas, showing that all three areas follow the same true curve initially, but as necking develops the other areas drop out of the deformation process.

Hexagonal cross sections of 10 mm height were cut at various elevations between +160 mm and –160 mm measured relative to the core center-plane. From these sections flat rectangular specimens of size 20×2×0.3 mm were mechanically produced. Subsequently, mini-tensile specimens with gauge length of 10x2x0.3 mm were produced by mechanical grinding and electrolytic polishing to achieve the desired dimensions and surface quality.

Pneumatic grips were used for holding the specimen in an Instron-1195 tensile machine. Uniaxial tensile tests on both unirradiated and irradiated specimens were performed at 20ºC at a strain rate of 8.3 x 10^{-4} sec^{-1}.

A technique called “digital marker extensometry” was used which incorporates digital video recording of the specimen during deformation. The surface of the specimen was marked with small (~0.3 mm) dots of dye in order to track the deformation on a local level. This technique was described in an earlier report and is especially useful in observing highly-irradiated miniature specimens subject to intense flow-localization [1, 2]. Application of this technique makes it possible to obtain the “true stress–true strain” behavior for a miniature specimen, as well as to identify the localized deformation region and to trace its evolving geometry during continuous deformation.

Results

In Figure 3 the engineering diagrams of both irradiated and unirradiated specimens are shown. As expected the unirradiated steel is characterized by high ductility and high ability to strain harden. Following irradiation to ~15 dpa at 340ºC the yield strength of a similar steel strongly increases and a neck develops just after reaching the yield point. The uniform elongation is very small and total ductility falls to 3.7%.

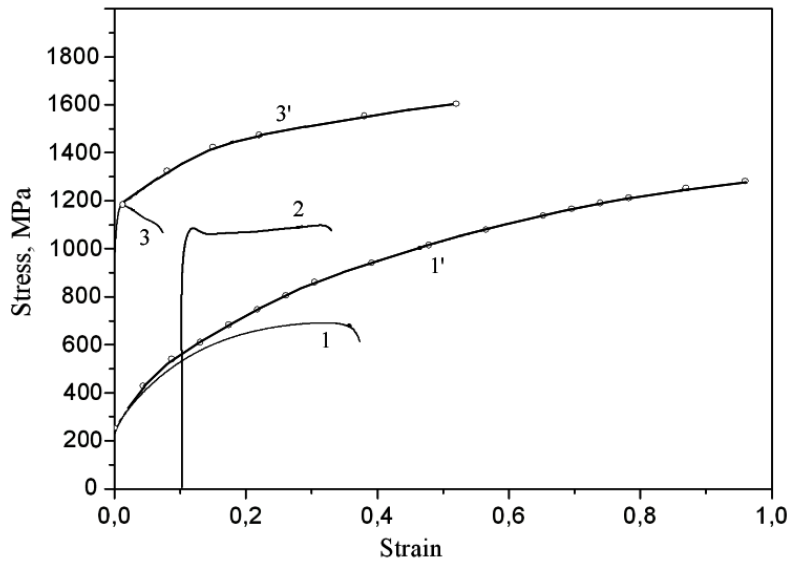

Figure 3. Engineering diagrams (curves 1, 2, 3) at 20ºC for 12Cr18Ni10Ti (1 and 2) and 08Cr16Ni11Mo3 irradiated in BN-350 (3) along with the corresponding “true” curves (1’ and 3’): 1 – unirradiated steel; 2 –irradiated to 55 dpa; 3 –irradiated to 15.6 . Curve 2 is shifted to the right to make visualization easier.

Based on current perceptions of saturation, one would expect that steel irradiated up to 55 dpa would achieve deformations <3.7%, even in the absence of void swelling. However, a ductility of 35 to 40% was achieved in this specimen. This result was confirmed by other tests to be typical and not an anomaly. Note that after a small decrease in strength after yielding there is an extended plateau without significant increase in load.

As shown in Figure 4, a series of freeze-frame video images taken during tensile testing shows that localized deformation initially forms near the upper gripp position, most likely due to stress concentration by the gripp. However, in contrast to irradiation to lower doses, a neck did not develop at 55 dpa. The localized deformation band instead progressively extended its lower boundary, producing a moving deformation front or deformation wave that moved down the specimen. The wave moved along ~2/3 of the specimen length. All of the deformation at a given instant appeared to occur at the wave front with material behind or in front of the wave being very weak or nonexistent.

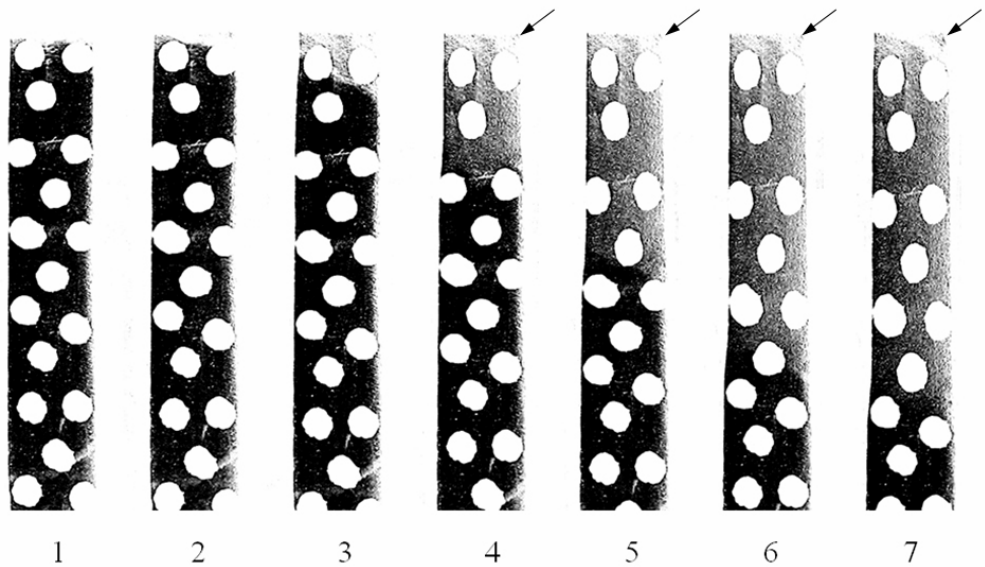

Figure 4. Freeze frames taken during deformation at 20ºc of the specimen irradiated to 55 dpa. Photographs have been digitally processed to increase the contrast. The boundary between the lighter distorted and darker undistorted areas moves downward with time. Distorted dots behind the boundary also show the local distortion. Arrows on photos 4 through 7 show the second later-developing and immobile neck.

Figure 5 shows the distribution of local deformation over the specimen length as the test progresses. An abrupt increase in local deformation from zero up to 30 to 35 % is observed at the moment the front passes that point. Failure with local deformation exceeding 60% occurred very near to the original place where the deformation wave appeared. It appears that the original wave might have continued its downward progress if failure had not occurred near the top of the specimen. Otherwise, formation of this second front had no effect on the progress of the original wave.

Failure arose via development of a second late-developing deformation band that was immobile. This band is marked by arrows on frames 4 through 7 in Figure 4. The immobility of the second deformation band was probably a result of the constraints imposed on the band by both the upper gripper and the upper boundary of the original wave. This suggests that deformation within the

deformed band is terminal and will not allow the second band to propagate through already deformed material.

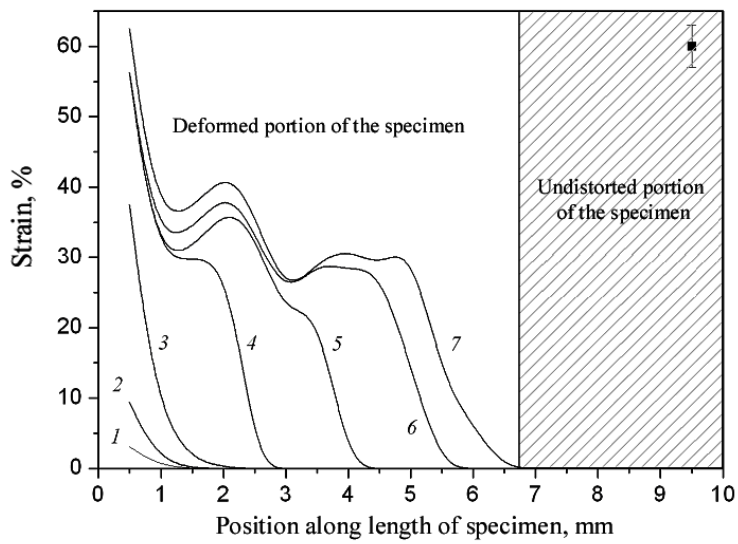

Figure 5. Distribution of local deformation along the length of the specimen irradiated to 55 dpa at various stages of the experiment. Figures 1 through 7 correspond to numbers of photographs in Figure 4.

Discussion

The condition for occurrence and development of localized deformation of the neck is [8, 9]

$$d\sigma/d\varepsilon \leq \sigma, \tag{1}$$

which can be rewritten in more convenient form:

$$d\sigma/d\varepsilon - \sigma \leq 0 \tag{2}$$

One may show that localization of deformation in compliance with a given condition starts at the moment when local strain hardening can no longer compensate for geometrical “softening” which occurs as a result of a decrease in the specimen cross section.

It’s clear that for stopping of local neck formation and displacement of the deformation into neighboring, less deformed space, the law which governs hardening must be changed, i.e., it is necessary that relation (2), on achieving a certain extent of deformation, becomes invalid. As a rule this does not happen in either unirradiated or neutron-irradiated pure metals, where $d\sigma/d\varepsilon$ always decreases as ε grows (see for example curve 1 on Figure 6).

Figure 6 presents “stress-deformation” curves obtained using the marker extensometry technique. One may observe (see curve 3’ Figure1 and curve 2 Figure6) that in 12Cr18Ni10Ti at 55 dpa the initial stage of deformation is close to that of 08Cr16Ni11Mo3 at ~15 dpa. Almost immediately on reaching the yield point, $d\sigma/d\varepsilon$-σ reduces to negative values, and the neck develops. However, in contrast to other materials we have studied, at local deformations of ~25 to 30% a smooth upward trend is observed in the “σ-ε” curve. As $d\sigma/d\varepsilon$ increases and the value of “$d\sigma/d\varepsilon$-σ” becomes positive, indicating that strain hardening is increasing strongly.

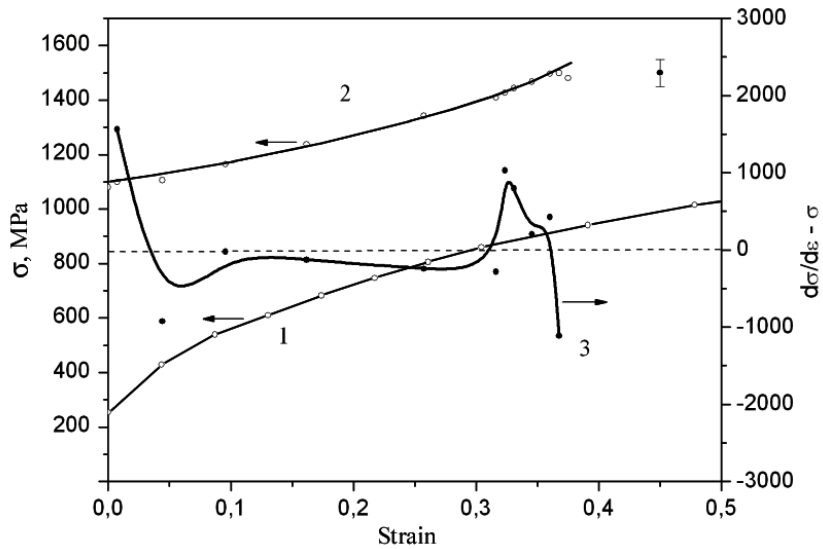

Figure 6. Curves of "true stress–true strain" for unirradiated 12Cr8Ni10Ti specimen (1) and 55 dpa specimen (2). The dependence of ($d\sigma/d\varepsilon$ - σ) versus ε (for 55dpa-sample) is shown in curve(3).

Apparently it is the increase in $d\sigma/d\varepsilon$ that leads to suppression of development of a local neck, thereby displacing the deformation source to neighboring, undeformed space, thus generating the deformation wave. We consider it to be very significant that the second late-forming deformation band could not move through the previously deformed region.

One potential source of the wave phenomenon is the $\gamma\rightarrow\alpha$ martensitic transformation. This low-nickel steel is known to be very sensitive to martensite formation, especially during low temperature deformation, and to increase in propensity toward martensite with radiation-induced hardening and radiation-induced segregation [10]. The fact that this behavior occurs at higher exposures but not at lower doses where saturation of strength had already occurred was at first thought to reflect some second-order effect such as the progressive transmutation-induced loss of Mn, one of the γ-stabilizing elements. A significant loss of the Mn due to transmutation has now been ruled out, with calculations showing only 2-3% maximum loss. Therefore some other mechanism is being sought, perhaps one related to radiation-induced segregation.

Similar deformation behavior involving an increase in intensity of strain hardening has been observed in this same steel in the unirradiated condition during deformation at cryogenic temperatures [11]. Intense martensitic transformation was cited as the cause, but the marker extensometry technique wasn't used in this experiment so it is not certain whether a deformation wave was associated with this behavior.

Conclusions

A new radiation-induced phenomenon has been observed in steel 12Cr18Ni10Ti irradiated to 55 dpa. It involves "a moving wave of plastic deformation" at 20ºC that produces "anomalously" high values of engineering ductility, especially when compared to deformation occurring at lower neutron exposures or for more stable steels. Using the technique of digital optical extensometry the "true stress σ –true strain ε" curves were obtained. It was shown that a moving wave of plastic deformation occurs as a result of an increase in the strain hardening, $d\sigma/d\varepsilon(\varepsilon)$. The increase in strain hardening is thought to arise from an irradiation-induced increase in the propensity of the $\gamma\rightarrow\alpha$ martensitic transformation

Acknowledgements

The Kazakh portion of this work was supported by the Ministry of Energy and Mineral Resources of the Republic of Kazakhstan. The US portion was sponsored by the Office of Fusion Energy, US Department of Energy.

References

[1] O. P. Maksimkin, M. N. Gusev, I. S. Osipov, “Deformation extensometry during mechanical tests of highly radioactive metal and alloy specimens”, Information News of the National Nuclear Center of the Republic of Kazakhstan, 2005, Issue 1, pp. 46-52.
[2] M. N. Gusev, O. P. Maksimkin, I. S. Osipov and F. A. Garner, “Application of Digital Marker Extensometry to Determine the True Stress-Strain Behavior of Irradiated Metals and Alloys”, Proceedings of 5th International Symposium on Small Specimen Test Technology”, in press.
[3] F. A. Garner, M. L. Hamilton, N. F. Panayotou and G. D. Johnson, "The Microstructural Origins of Yield Strength Changes in AISI 316 During Fission or Fusion Irradiation," J. Nucl. Mater., 103 and 104 (1981), 803-808.
[4] F. A. Garner, Chapter 6: "Irradiation Performance of Cladding and Structural Steels in Liquid Metal Reactors," Vol. 10A of Materials Science and Technology: A Comprehensive Treatment, VCH Publishers, 1994, pp. 419-543.
[5] M. L. Hamilton, F. H. Huang, W. J. S. Yang and F. A. Garner, "Mechanical Properties and Fracture Behavior of 20% Cold-Worked 316 Stainless Steel Irradiated to Very High Exposures," Effects of Radiation on Materials: Thirteenth International Symposium (Part II) Influence of Radiation on Material Properties, ASTM STP 956, F. A. Garner, N. Igata and C. H. Henager, Jr., Eds., ASTM Philadelphia, PA, 1987, pp. 245-270.
[6] A. M. Parshin, “Structure Resistibility and Radiation Damage in Corrosion-resistant Steels and Alloys”, Chelyabinsk, “Metallurgy”, Chelyabinsk Department, 1988, p. 656 (in Russian).
[7] V. Barabash, G. Kalinin et al., “Materials Challenges for ITER – Current Status and Future Activities”, J. Nucl. Mater. 367-370 (2007) pp. 21-32.
[8] G. A. Malygin, “Analysis of structural factors responsible for formation of the bottle neck area during tensile tests of fcc metals and alloys”, Solid State Physics 47, Issue 2 (2005) 236-241.
[9] V. I. Trefilov, V. F.Moyiseev and others, “Deformation hardening and failure of polycrystal metals”, Kiev, “Naukova Dumka”, 1989, 256 p..
[10] G. Was “Fundamentals of Radiation Materials Science. Metals and Alloys” “Springer”, 2007. E. Nagy, V. Mertinger, F. Tranta, J. Sólyom, “Deformation induced martensitic transformation in stainless steels”, Materials Science and Engineering A 378 (2004) 308–313.