Journal of Nuclear Materials 386–388 (2009) 273–276

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Anomalously large deformation of 12Cr18Ni10Ti austenitic steel irradiated to 55 dpa at 310 °C in the BN-350 reactor

M.N. Gusev [a,*], O.P. Maksimkin [a], I.S. Osipov [a], F.A. Garner [b]

[a] Institute of Nuclear Physics, Almaty, Kazakhstan
[b] Pacific Northwest National Laboratory, Richland, WA, USA

ABSTRACT

Whereas most previous irradiation studies conducted at lower neutron exposures in the range 100–400 °C 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 20 °C 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.

© 2009 Elsevier B.V. All rights reserved.

1. Introduction

It is generally accepted that irradiation of stainless steels at temperatures of 100–400 °C 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]. Figs. 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 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.

2. Experimental details

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 15–20%, 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 280 °C defines the lowest temperature of the wrapper.

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 10 × 2 × 0.3 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 20 °C at a strain rate of $8.3 \times 10^{-4}\ s^{-1}$.

A technique called 'digital marker extensometry' was used which incorporates digital photo or video recording of the specimen during deformation. The surface of the specimen was marked

* Corresponding author.
E-mail address: gusev.maxim@inp.kz (M.N. Gusev).

0022-3115/$ - see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2008.12.115

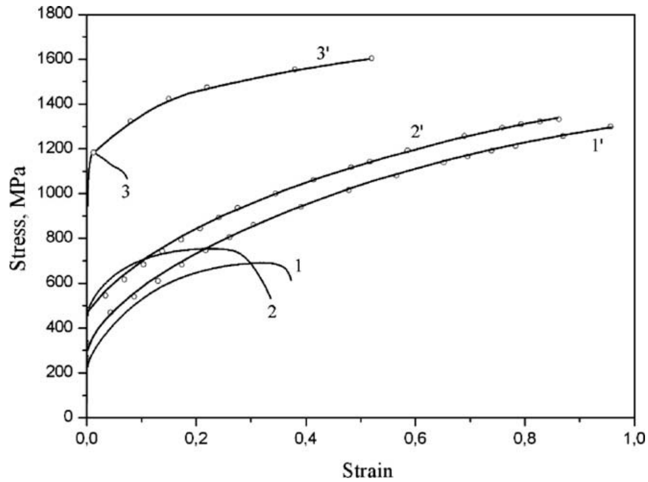

Fig. 1. Engineering stress–strain curves (1, 2, 3) and 'σ_i–ε_i' relationships (1′, 2′, 3′) for non-irradiated (1) and irradiated (2, 3) stainless steels tested at 20 °C. 2, 12Cr18Ni10Ti (WWR-K, 1.4 × 10^{19} n/cm^2 and 80 °C); 3, 08Cr16Ni11Mo3 (BN-350, 15.6 dpa and 340 °C).

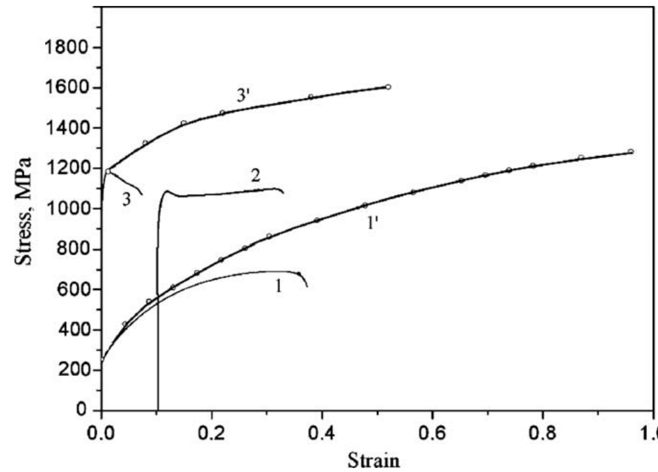

Fig. 3. Engineering stress–strain diagrams (curves 1, 2, 3) at 20 °C for 12Cr18Ni10Ti (1) and (2) and 08Cr16Ni11Mo3 irradiated in BN-350 (3) along with the corresponding 'true' curves (1′ and 3′): 1, unirradiated steel; 2, irradiated to 55 dpa; 3, irradiated to 15.6. Curve 2 is shifted to the right to make visualization easier.

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.

3. Results

In Fig. 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 340 °C 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%.

Based on current perceptions of saturation, one would expect that steel irradiated up to 55 dpa would achieve deformations <7.5%, even in the absence of void swelling. However, a ductility of 20–25% was achieved in this specimen. This result was confirmed by ten other tests to be typical and not an anomaly. Note that after a small decrease in strength following yielding there is an extended plateau without significant increase in load.

As shown in Fig. 4, a series of freeze-frame video images taken during tensile testing shows that localized deformation initially forms near the upper grip position, most likely due to stress concentration by the grip. 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 deformation behind or in front of the wave being very weak or nonexistent.

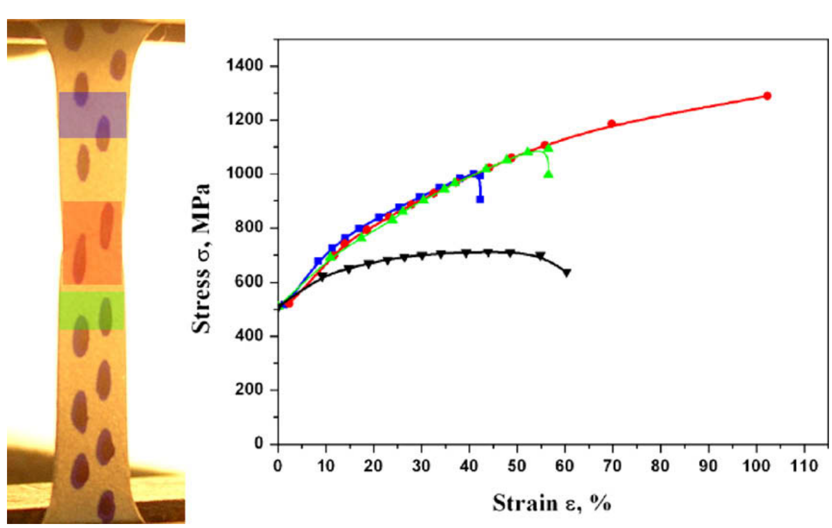

Fig. 2. 12Cr18Ni10Ti irradiated to 1.4 × 10^{19} n/cm^2 (E > 0.1) in the WWR-K reactor at 80 °C and tested at 20 °C. Comparison of engineering (black) and true strain curves (designated by colors) for three areas, showing that all three areas follow the same true curve initially, but as necking develops the other areas drop out of the deformation process. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

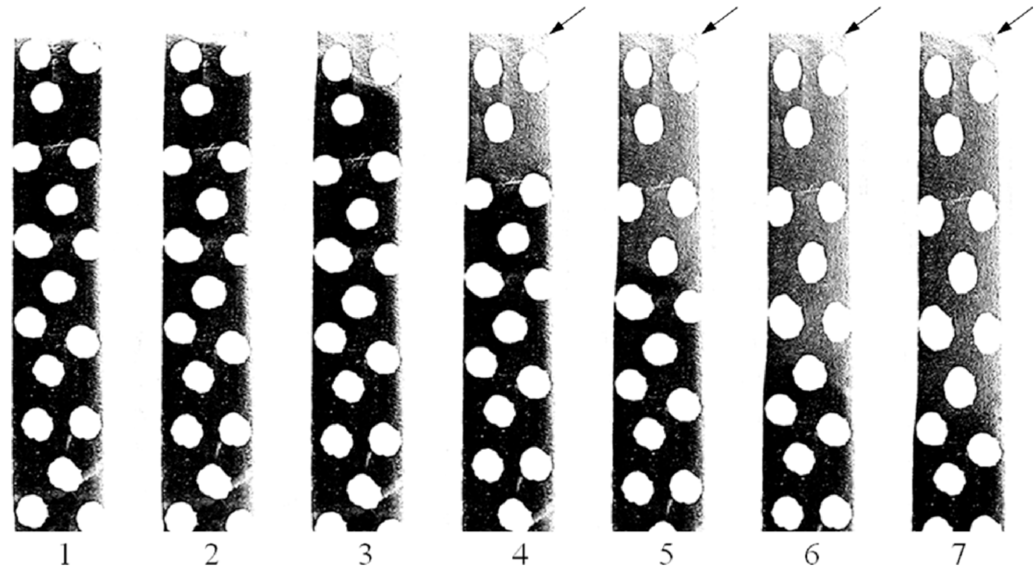

Fig. 4. Freeze frames taken during deformation at 20 °C of the specimen irradiated to 55 dpa. Photographs have been digitally processed to increase the contrast. The boundary between the lighter distorted and darker undistorted areas moves downward with time. Distorted dots behind the boundary also show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

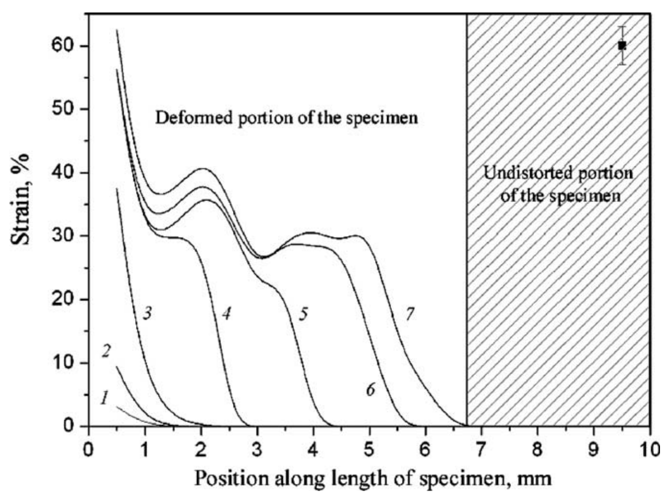

Fig. 5. Distribution of local deformation along the length of the specimen irradiated to 55 dpa at various stages of the experiment. Curves 1 through 7 correspond to numbers of photographs in Fig. 4.

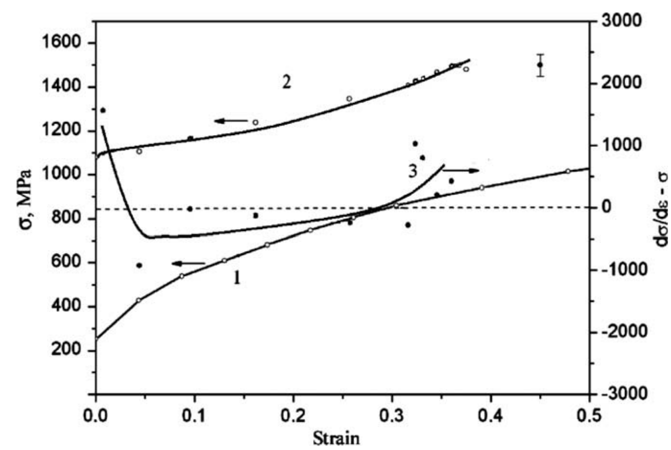

Fig. 6. Curves of 'true stress–true strain' for unirradiated 12Cr8Ni10Ti specimen (1) and 55 dpa specimen (2). The dependence of $(d\sigma/d\varepsilon - \sigma)$ versus ε (for 55 dpa-sample) is shown in curve (3).

Fig. 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 30–35% 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 Fig. 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.

4. Discussion

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

$$d\sigma/d\varepsilon \le \sigma, \quad (1)$$

which can be rewritten in more convenient form:

$$d\sigma/d\varepsilon - \sigma \leqslant 0. \quad (2)$$

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 is 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 Fig. 6).

Fig. 6 presents 'stress-deformation' curves obtained using the marker extensometry technique. One may observe (see curve 3′ Fig. 1 and curve 2 Fig. 6) 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, $d\sigma/d\varepsilon - \sigma$ reduces to negative values, and the neck develops. However, in contrast to other materials we have studied, at local deformations of ~25–30% a smooth upward trend is observed in the 'σ–ε' curve. As $d\sigma/d\varepsilon$ increases the value of '$d\sigma/d\varepsilon - \sigma$' becomes positive, indicating that strain hardening is increasing strongly.

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 and this possibility is now being investigated. 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 first thought to reflect some second-order effect such as the progressive transmutation-induced loss of Mn, one of the γ-stabilizing elements. However, calculations showed only 2–3% loss of Mn had occurred by 55 dpa. Therefore other mechanisms such as radiation-induced segregation are being investigated.

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 was not used in this experiment so it is not certain whether a deformation wave was associated with this behavior.

5. 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 20 °C 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, Informat. News National Nucl. Center Republic of Kazakhstan (1) (2005) 46.
[2] M.N. Gusev, O.P. Maksimkin, I.S. Osipov, F.A. Garner, Application of digital marker extensometry to determine the true stress–strain behavior of irradiated metals and alloys, in: Proceedings of 5th International Symposium on Small Specimen Test Technology, Paper 016803JAI, Journal of ASTM International, in press.
[3] F.A. Garner, M.L. Hamilton, N.F. Panayotou, G.D. Johnson, J. Nucl. Mater. 103&104 (1981) 803.
[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, 1994.
[5] M.L. Hamilton, F.H. Huang, W.J.S. Yang, F.A. Garner, Mechanical properties and fracture behavior of 20% cold-worked 316 stainless steel irradiated to very high exposures, in: F.A. Garner, N. Igata, C.H. Henager Jr. (Eds.), Effects of Radiation on Materials: Thirteenth International Symposium (Part II) Influence of Radiation on Material Properties, ASTM STP 956, ASTM, Philadelphia, 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., J. Nucl. Mater. 367–370 (2007) 21.
[8] G.A. Malygin, Solid State Phys. 47 (2) (2005) 236.
[9] V.I. Trefilov, V.F.Moyiseev, et al., Deformation hardening and failure of polycrystal metals, Kiev, Naukova Dumka, 1989, 256pp.
[10] G.S. Was, Fundamentals of Radiation Materials Science, Springer, 2007.
[11] E. Nagy, V. Mertinger, F. Tranta, J. Sólyom, Mater. Sci. Eng. A378 (2004) 308.