Manifestations of DSA in Austenitic Stainless Steels and Inconel Alloys

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1 ABSTRACT

Dynamic strain aging (DSA) affects mechanical properties of the materials and promotes strain localisation. DSA results in serrated plastic flow, which was observed by means of constant extension rate tensile (CERT) tests in commercial AISI 316NG austenitic stainless steel and Inconel 600 and Inconel 690 Ni-base alloys at temperature range of 200 – 600 °C and values of strain rate from 10\textsuperscript{3} to 10\textsuperscript{6} s\textsuperscript{-1}. Negative strain rate sensitivity, which is another manifestation of DSA, was also reported for the studied materials at temperatures and strain rates where serrated plastic flow appeared. The map for occurrence of serrated flow as a function of strain rate and temperature was built for these materials. The activation enthalpies of dynamic strain aging appearance were found to be 120 kJ/mol for the austenitic stainless steel and 159 kJ/mol for both Ni-base alloys. The internal friction (IF) peak associated with the interstitial atoms of nitrogen and carbon in the solid solution of AISI 316NG austenitic stainless steel and Inconel 600 alloy was reported. The enthalpies of nitrogen diffusion in AISI 316NG steel (140 kJ/mol) and carbon diffusion in Inconel 600 alloy (162 kJ/mol), obtained by means of internal friction, correspond well to the enthalpies of DSA appearance. The height of the internal friction peak increases depending on the pre-straining conditions in the similar way for AISI 316NG steel and Inconel 600 alloy. Annealing in situ at the IF peak temperature results in the decay of the IF peak enhanced by pre-straining for both materials. At the initial stage, the peak annealing processes can be described as an elemental exponential decay function with characteristic times of 0.95 and 0.92 ks for AISI 316NG steel and Inconel 600 alloy, respectively. Transmission electron microscopy was performed on the specimens of AISI 316NG steel after CERT tests at temperatures of 400 and 200 °C, where serrated and smooth plastic flow was observed, respectively. Long-range planarity was observed in the dislocation structures of the specimen tested at 400 °C. The microstructure of the specimen strained at 200°C exhibited cellular dislocation structure. It was concluded, that diffusion re-distribution of nitrogen in the DSA regime affected the deformation behaviour of the material by restricting cross-slip, which in turn promotes strain localization, degrading the mechanical performance of the material.

2 INTRODUCTION

Dynamic strain aging (DSA) is a material phenomenon when aging occurs during the deformation. DSA occurs in alloys containing solute atoms which can rapidly and strongly segregate to dislocations during straining. This phenomenon causes a remarkable degradation of mechanical properties and results in strain localization that can affect crack initiation and propagation. Known physical manifestations of DSA are as follows: serrated plastic flow (or abrupt changes in stress observed on stress-strain curves during deformation); negative strain rate sensitivity; blue brittleness; maximum in variation of ultimate tensile strength and strain hardening with temperature; and peak in the variation of Hall-Petch slope with temperature (Rodriguez 1984). Serrated plastic flow is one of the main well studied macroscopic manifestations of the DSA. The temperature of DSA occurrence is material dependent, e.g. for low-alloy RPV steel serrations are observed at 140-340 °C (Kim & Kang 1995), in austenitic stainless steels they occur in the range 300 to 650 °C (Kim et al. 1998; Hong & Lee 2005; Ehrnstén et al. 2005), while in Ni-base alloys serrated plastic flow was observed at the temperatures from 200 °C to 600 °C (Hänninen et al. 2005; Mulford & Kocks 1979).
At different temperatures various solute atoms and a number of mechanisms can participate in the solute atom – mobile dislocation interactions. The DSA, which is observed at temperatures up to 500 °C, manifests itself as serrated plastic flow of types A and B. Interaction of interstitials (C and N) or interstitial-vacancy pairs with dislocations during deformation is considered to be responsible for the DSA in austenitic stainless steels and Ni-base alloys at these temperatures. At temperatures about 600 °C, where the serrations of type C are observed, the DSA is attributed to interaction of substitutional solute atoms (Cr or Mo) with mobile dislocations (Kim et al. 1998; Hong & Lee 2004, 2005; Mulford & Kocks 1979; Ivanchenko et al. 2004; Ehrnstén et al. 2005, Hänninen et al. 2005; Chen & Chaturvedi 1997).

It was shown that in austenitic stainless steel cold-work prior to tensile testing promotes the appearance of serrations, while alloying with nitrogen shifts the temperature range of DSA observation to higher temperatures (Hong & Lee 2004; Kim et al. 1998; Ivanchenko et al. 2004).

In Ivanchenko et al. (2004) and Hanninen et al. (2005) the Fourier analysis of the stress serrations observed in AISI 316 NG austenitic stainless steel and Ni-base alloys Inconel 600 and Inconel 690 was performed and average values of time between stress pulses were estimated. It was shown that some of these estimated values correlate with characteristic times of nitrogen and carbon re-distribution in solid solution of the studied materials, which were obtained by means of internal friction.

As it is seen from above, interstitial atoms such as carbon and nitrogen play an important role in the mechanical properties of low-alloy RPV steel, austenitic stainless steels and Ni-base alloys. Strength, creep resistance and fatigue life of these alloys in the environmental conditions of nuclear reactors depend on the amount, state and diffusion mobility of interstitial atoms dissolved in the solid solution results in dynamic strain aging (DSA) of the steels and Ni-base alloys at temperatures above 200 °C (Ehrnstén et al. 2007; Hänninen et al. 2005; Mulford & Kocks 1979).

The aim of the present investigation was to examine and compare the DSA manifestations in AISI 316NG austenitic stainless steel (SS) and Ni-base alloys Inconel 600 and Inconel 690 by means of constant extension rate tensile (CERT) and step-wise tensile tests, mechanical loss spectrometry (internal friction) and transmission electron microscopy (TEM). Another aim was to determine differences in the resulting dislocation structures and internal friction response of the materials pre-strained under conditions promoting and not promoting DSA behaviour.

3 EXPERIMENTAL AND MATERIALS

Austenitic stainless steel AISI 316NG and two commercial Ni-base alloys Inconel 600 and Inconel 690 were studied. The chemical compositions of the materials are shown in Table 1. All the materials were supplied in mill-annealed state. Average grain size of the studied alloys was about 72, 100 and 30 µm for AISI 316NG steel, Inconel 600 and Inconel 690 alloys, respectively.

<table>
<thead>
<tr>
<th>Type</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>C</th>
<th>N</th>
<th>Ti</th>
<th>Mo</th>
<th>Co</th>
<th>Mn</th>
<th>Al</th>
<th>Cu</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>AISI 316NG</td>
<td>bal.</td>
<td>12.5</td>
<td>17</td>
<td>0.022</td>
<td>0.093</td>
<td>2.28</td>
<td>1.66</td>
<td>0.01</td>
<td>0.11</td>
<td>0.38</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inconel 600</td>
<td>8.1</td>
<td>bal.</td>
<td>16.1</td>
<td>0.04</td>
<td>-</td>
<td>0.16</td>
<td>0.29</td>
<td>0.22</td>
<td>0.17</td>
<td>0.01</td>
<td>0.32</td>
<td></td>
</tr>
<tr>
<td>Inconel 690</td>
<td>8.4</td>
<td>bal.</td>
<td>27.7</td>
<td>0.01</td>
<td>-</td>
<td>0.14</td>
<td>0.07</td>
<td>0.22</td>
<td>0.18</td>
<td>0.01</td>
<td>0.23</td>
<td></td>
</tr>
</tbody>
</table>

All tensile test specimens were prepared according to ASTM standard E8M (sheet-type sub-size specimens). Tensile tests were performed according to the standards SFS-EN 1002-1 and ASTM E21 (Standard Test Method for Elevated Temperature Tension Tests of Metallic Materials) in air environment. Tensile tests were carried out using a 25 kN MTS 858 test machine equipped with a MTS High-Temperature Furnace 653.02 at strain rates in the range of \(10^{-5} - 10^{-6}\) s\(^{-1}\) and temperatures from 100 to 800 °C. The strain rate sensitivity was measured using step-wise changes of the strain rate from \(10^{-5}\) to \(10^{-6}\) s\(^{-1}\) and in the reverse
way during tensile testing, with the ratio of initial and subsequent strain rate \((\dot{\varepsilon}_1/\dot{\varepsilon}_2)\) equal to 100 or 0.01. To calculate the strain rate sensitivities in true stress – true strain coordinates a linear interpolation of each strain rate interval was performed. The step-wise tests were performed at 200, 288 and 400 °C for AISI 316NG steel and at 100, 200 and 300 °C for Inconel 600 and Inconel 690 alloys.

The internal friction (IF) method was used to indicate the presence of the free nitrogen and carbon and their diffusive redistribution in the crystalline lattice of the studied materials. Specimens of AISI 316NG austenitic stainless steel for IF measurements were taken from the gauge lengths of tensile test specimens strained to 20 % of elongation at strain rate of \(10^{-3}\) s\(^{-1}\) and temperatures of 200, 288 and 400 °C. The IF specimens of Inconel 600 and Inconel 690 alloys were taken from tensile test specimens strained to about 50 % of elongation at strain rate of \(10^{-3}\) s\(^{-1}\) and temperatures of 100, 200 and 300 °C (Hänninen et al. 2005). The cooling time to 26 °C after the straining at elevated temperatures did not exceed 2 min for all samples used for further IF measurements. The specimens had the typical dimensions of 0.5 × 1.5 × 45 mm, and were prepared by cutting with an abrasive disc saw and then mechanically polishing with 1200 grit emery paper.

The temperature dependencies of the internal friction, \(Q^1\), were measured using the method of free decay of resonance oscillation by an inverted torsion pendulum over the temperature range from 28 to 500 °C with the amplitudes of the torsion deformation less then \(10^{-5}\). The natural frequency of the pendulum was ~1.5 Hz. The heating rate was 2 °C/min.

TEM examinations were performed only for AISI 316NG austenitic stainless steel. Specimens for TEM examination were prepared from the deformed gauge region of tensile specimens tested to fracture at strain rate of \(10^{-3}\) s\(^{-1}\) and temperatures of 200 and 400°C. The cooling time of the tensile specimens to 26 °C after the straining at elevated temperatures did not exceed 2 min. Because of the thin sheet geometry of the tensile specimens, they were first ground to <0.1 mm thickness, and then 3 mm diameter disk specimens for TEM were punched from the fractured bars in the plane orientation. The disks were subsequently electropolished to electron transparency with a Tenupol 5 running a 3% perchloric acid/methanol solution at -35 °C and 23-26 V.

4 RESULTS AND DISCUSSION

4.1 Mechanical manifestations of DSA

Manifestations of DSA by serrated flow and negative strain rate sensitivity for studied materials are shown in Figs. 1 – 3.

The development of serrated flow with temperature of the tensile test conducted at strain rate of \(10^{-3}\) s\(^{-1}\) for AISI 316NG austenitic stainless steel is shown in Fig. 1a. The tensile tests revealed that in AISI 316NG austenitic stainless steel serrated flow is well-defined at the testing temperatures from 288 to 500 °C, while at 200 °C and above 600 °C serrated flow was not observed. At 288 °C only periodic abrupt changes in stress, which correspond to serrated flow of type A were present on the stress-strain curve. In the test conducted at 400 °C, the amplitude of the serrations increased with strain and they changed in series from A to A+B type, and at higher strain values, to mainly B-type serrations, which are oscillations about general level of the stress-strain curve that occur in quick succession. At 500 °C only B-type serrations were observed. Dependences of strain rate sensitivity from temperature of step-wise tensile test and flow stress for AISI 316NG steel are shown in Fig. 1b. The results obtained for strain rate sensitivity correlate with the development of serrated flow. At 200 °C, while serrations are not observed on stress-strain curve, the strain rate sensitivity remains positive for all the values of flow stress. At 288 °C, while only serrations of type A were observed, strain rate sensitivity was about zero with minor deviations to negative values at flow stresses over 400 MPa. At 400 °C, when the serrated flow is well developed and amplitude of the serrations is the maximal, the strain rate sensitivity had negative values already at 200 MPa and was decreasing with increasing flow stress.

The temperature range, where the serrated flow is observed, is wider for Inconel 600 and Inconel 690 alloys than for AISI 316NG steel, strained at the same strain rate of \(10^{-3}\) s\(^{-1}\). Serrated flow of type A becomes well recognised on stress-strain curves obtained at strain rate of \(10^{-3}\) s\(^{-1}\) already at 150 °C for Inconel 600 alloy, Fig. 2a, and at 200 °C for Inconel 690 alloy. At 300 and 400 °C serrated flow changes to B-type and at 600 °C to type C for both studied Ni-base alloys. The measurements of strain rate sensitivity for these alloys are shown in Figs. 2b and 3b. As can be seen from Figs. 2 and 3, positive strain rate sensitivity values
correspond to deformation conditions where smooth flow was observed. At 200 and 300 °C serrated flow appears on stress-strain curves and strain rate sensitivity values become negative.

The simultaneous observation of serrated flow and negative strain rate sensitivity is a clear confirmation of DSA phenomenon in the studied materials.

Figure 1. Manifestations of DSA in AISI 316NG austenitic stainless steel on engineering stress-strain curves obtained at strain rate of $10^{-5}$ s$^{-1}$ (a) and on dependences of strain rate sensitivity on flow stress obtained from step-wise tests at strain rates of $10^{-4}$ and $10^{-6}$ s$^{-1}$ (b).

Figure 2. Manifestations of DSA in Inconel 600 alloy on engineering stress-strain curves obtained at strain rate of $10^{-5}$ s$^{-1}$ (a) and on dependences of strain rate sensitivity on flow stress obtained from step-wise tests at strain rates of $10^{-4}$ and $10^{-6}$ s$^{-1}$ (b).
Dependences of the onset of serrations in AISI 316NG steel, Inconel 600 and Inconel 690 alloys on temperature and strain rate are shown in Fig. 4. The data summarises the appearance of serrations on the stress-strain curves of the studied materials at different strain rates and testing temperatures. In the case of filled data points DSA serrations take place while open ones mean that no serrations were observed. The dotted lines in Fig. 4 represent dependences of the critical strain rate as a function of testing temperature for DSA occurrence. This dependence is widely used for calculation of enthalpy of DSA occurrence and it can be expressed as

\[ \dot{\varepsilon}_c = \dot{\varepsilon}_c^0 \exp \left( -\frac{H}{k_B T} \right), \]

where \( \dot{\varepsilon}_c^0 \) is a pre-exponential factor, \( k_B T \) is temperature in energy units and \( H \) is representing the apparent enthalpy of the DSA occurrence. The enthalpies for the onset of DSA calculated from the dotted lines in Fig. 4 are about 120 kJ/mol for AISI 316NG steel and about 159 kJ/mol for both Ni-base alloys. The boundary for Inconel 600 alloy is at lower temperatures as compared to that of Inconel 690 alloy.

**Figure 3.** Manifestations of DSA in Inconel 690 alloy on engineering stress-strain curves obtained at strain rate of 10^{-5} s^{-1} (a) and on dependences of strain rate sensitivity on flow stress obtained from step-wise tests at strain rates of 10^{-4} and 10^{-5} s^{-1} (b).

**Figure 4.** Comparison maps of the serrated flow appearance depending on temperature and strain rate for AISI 316NG steel and Inconel 600 alloy shown by grey circles and red squares, respectively (a) and for Inconel 600 and Inconel 690 alloys shown by grey squares and blue circles, respectively (b). Filled data points correspond to strain rate and temperature values at which serrated flow occurs on stress-strain curves and open points correspond to smooth flow. The dashed lines are the boundaries for the onset (or disappearance) of serrations.

### 4.2 Interstitial atoms distribution

The IF peak observed in AISI 316NG austenitic SS and Inconel 600 alloy with maxima in the vicinity of 350 °C was recognised as Snoek-like relaxation process (Ivanchenko et al. 2004, 2006). This relaxation process is induced by re-orientation of interstitial atoms in FCC lattice of austenitic stainless steels under periodically applied external stresses originally reported by Rosin and Finkelstein (1953) and was confirmed by numerous authors in different FCC alloys and pure metals (Blanter et al. 2007). It has been established that the height of the Snoek-like peak is proportional to concentration of interstitial atoms in solid solution of the steels with FCC lattice (Verner 1965). The peak height and its temperature position may vary depending on the chemical composition of the FCC alloys and depend on distribution of interstitial atoms in the lattice. The corresponding studies and the model for this phenomenon were presented by Grujicic et al. (1988), Grujic & Owen (1995), Jagodzinski et al. (1998) and Yagodzinskyy et al. (2009). The model suggested in these works was used to explain absence of Snoek-like peak in Inconel 690 alloy, while the DSA phenomenon was manifested (Ivanchenko et al. 2006).
The enthalpies of nitrogen diffusion in FCC lattice of AISI 316NG steel (140 kJ/mol) and enthalpy of carbon diffusion in the lattice of Inconel 600 alloy (162 kJ/mol) obtained by IF method are in good agreement with the apparent enthalpies of DSA occurrence (Ivanchenko et al. 2004, 2006). Observed correspondence of the activation enthalpies evidences that the appearance of the DSA results from the interaction of the free interstitial atoms (C and N) in the solid solution of the materials with mobile dislocations.

To examine the effect of the DSA on the re-distribution of carbon in Inconel 600 alloy and nitrogen in AISI 316NG austenitic SS, three different regimes of prior deformation at elevated temperatures were applied. For the first regime, the temperature of prior plastic straining was selected below the DSA occurrence, which is 100 °C for Inconel 600 alloy and 200 °C for AISI 316NG austenitic SS. For the second regime, temperature was selected in the way that the DSA was manifested by serrated flow of type A (200 and 288 °C for the Inconel 600 alloy and AISI 316NG austenitic SS, respectively). The third regime represents deformation under the DSA conditions, when the jerky flow of type B was observed. It corresponds to 300 °C for Inconel 600 alloy and 400 °C for AISI 316NG austenitic SS.

AISI 316NG austenitic SS and Inconel 600 alloy show the same evolution of Snoek-like peak after the prior deformation at elevated temperatures. Temperature dependencies of the internal friction of AISI 316NG austenitic SS and Inconel 600 alloy are shown in Fig. 5.

![Figure 5](image_url)

**Figure 5.** Effect of pre-straining at different temperatures with strain rate of $10^{-5}$ s$^{-1}$ on evolution of the Snoek-like internal friction peak in AISI 316NG austenitic stainless steel strained to 20% of elongation (a) and in Inconel 600 alloy strained to 50% of elongation (b). The background is subtracted.

Well-defined maxima on internal friction spectra are already apparent for the studied alloys in as-supplied condition. Pre-straining of the materials at elevated temperatures, which do not induce the DSA, increases the height of the Snoek-like peak in both studied alloys by about 3.5 times in relation to the corresponding as-supplied condition values. The effect was greater after the prior deformation at the temperatures, when the serrated yielding attributed to the DSA was observed. Prior deformation under the DSA condition manifested by serrated flow of type A led to more than seven-fold increase in peak height. The peak height was further elevated (about 10 times) after the pre-straining of the studied materials at temperatures, which induce the DSA serrations of type B.

The enhanced Snoek-like peaks obtained after pre-straining at DSA conditions are unstable. The peak amplitude decays with annealing at the peak temperature as shown in Fig. 6. At the initial stage (about 20 min), the peak annealing process can be described by an elemental exponential decay function (shown by dashed lines in Fig. 6) with characteristic decay time of $0.92 \pm 0.07$ ks and $0.95 \pm 0.07$ ks for Inconel 600 alloy and AISI 316NG austenitic SS, respectively.
Figure 6. Snoek-like peak amplitude decays with annealing time of 20 min at 347 and 365 °C for Inconel 600 alloy and AISI 316NG austenitic stainless steel, respectively. The decays fit well by exponential decays with characteristic times $\tau$ shown in the plot.

Increase in the peak height is related to atomic re-ordering during deformation and the formation of interstitial enriched sub-micron zones dragged by dislocation pile-ups during the DSA (Ivanchenko et al. 2006). Similar evolution of the Snoek-like peak with pre-straining at elevated temperatures and nearly the same values of characteristic times of the peak decays suggests the same mechanisms involved in atomic re-ordering in both studied materials, which occurs during the DSA.

4.3 Microstructural manifestations

TEM examination of AISI 316NG steel presented in Fig. 7 reveals the differences in dislocations substructures of the steel strained at two different temperatures at strain rate of $10^{-5}$ s$^{-1}$: 200 °C, while smooth flow was observed; and 400 °C, while DSA was manifested by jerky flow of type A+B. As shown in Fig. 7a, the microstructure in the material following tensile testing at 200 °C was comprised of small, thick-walled cells. Cellularization is a result of mutual annihilation of dislocations, and the reorganization of remaining dislocations into a lower energy structure. It is enabled by cross-slip, i.e. a dislocation gliding on multiple planes simultaneously. As shown in Fig. 7b, the microstructure following tensile testing at 400 °C showed much more linearity than that at 200 °C. That is a consequence of dislocation glide occurring on particular planes. According to DSA theory, the interaction between dislocations and solute atoms limits the freedom of motion of the dislocations; i.e. by pinning portions of the dislocations, the dislocations’ ability to cross-slip is restricted. That produces planar slip, which is manifested as linear microstructures.
Figure 7. TEM examination of AISI 316NG steel strained to fracture at strain rate of $10^5$ s$^{-1}$. The microstructure from the gauge region following tensile testing at 200 °C (beam is parallel to [100]) (a) and at 400 °C, which shows much more linearity than that at 200 °C (beam is parallel to [110]) (b). Arrows indicate g-vectors.

A comparison of the microstructures of the samples strained at 200 and 400 °C reveals some differences. In particular, the material tensile tested at 400 °C showed a greater tendency towards dislocation planarization, manifested as highly linear dislocation structures. That tensile test had also shown serrations on the tensile stress-strain curve, and a clearly increased Snoek-like peak height. On the other hand, the 200 °C tensile tested steel did not have such straight dislocations, nor had it shown serrated flow, while the Snoek-like peak was not as high as that for 400 °C. Thus, it would appear that the planarization in the dislocation microstructure correlates with the IF results, and both are correlated with the altered tensile stress-strain behaviour observed.

5 CONCLUSION

It was shown that DSA is present in commercial AISI 316NG steel and Inconel 600 and Inconel 690 alloys, and occurs at temperatures relevant to nuclear power plant operation.

The enthalpies of nitrogen and carbon diffusion in AISI 316NG steel and Inconel 600 alloy obtained by means of internal friction correspond well to the enthalpies of DSA appearance. Observed correspondence of the activation enthalpies evidences that the appearance of the DSA results from the interaction of the free interstitial atoms (carbon and nitrogen) in the solid solution of the materials with mobile dislocations.

Similar evolution of observed IF peak with pre-straining at elevated temperatures and nearly the same values of characteristic times of the peak decays suggests that the same mechanisms are involved in atomic re-ordering in the studied materials, which occur during the DSA. Dependency of the amplitude of the IF peak on regime of pre-straining is probably related to atomic re-distribution during plastic deformation and to the formation of interstitial enriched sub-micron zones dragged by dislocation pile-ups during the DSA. Dependence of the nitrogen- and carbon-induced IF-peak decays on time of annealing at the peak temperature reflects the post-pre-straining re-distribution of interstitials.

Diffusion re-distribution of nitrogen in the DSA regime affected the deformation behaviour of the material by restricting cross-slip therefore promoting strain localization and degrading the mechanical performance of the material.

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