Microstructure Evolution During Creep of Cold Worked Austenitic Stainless Steel

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Abstract: The 14Cr–15Ni austenitic stainless steel (SS) with additions of Ti, Si, and P has been developed for their superior creep strength and better resistance to void swelling during service as nuclear fuel clad and wrapper material. Cold working induces defects such as dislocations that interact with point defects generated by neutron irradiation and facilitates recombination to make the material more resistant to void swelling. In present investigation, creep properties of the SS in mill annealed condition (CW0) and 40 % cold worked (CW4) condition were studied. D9I stainless steel was solution treated at 1333 K for 30 minutes followed by cold rolling. Uniaxial creep tests were performed at 973 K for various stress levels ranging from 175-225 MPa. CW4 samples exhibited better creep resistance as compared to CW0 samples. During creep exposure, cold worked material exhibited phenomena of recovery and recrystallization wherein new strain free grains were observed with lesser dislocation network. In contrast CW0 samples showed no signs of recovery and recrystallization after creep exposure. Partial recrystallization on creep exposure led to higher drop in hardness in cold worked sample as compared to that in mill annealed sample. Accelerated precipitation of carbides at the grain boundaries was observed during creep exposure and this phenomenon was more pronounced in cold worked sample.

1. Introduction
Advanced nuclear power plants are being designed with the potential to fulfill rising future energy demands in an ecofriendly manner. The prime parameters which are dependent on the material performance are safety, reliability, and economical viability [1]. Austenitic stainless steels are candidate material for power plants as a structural component. In nuclear plants, fuel clad material requires better creep strength and resistance to irradiation-induced void swelling during service. These clad tubes are generally exposed to high temperature in the range of 673 to 973 K. Therefore, it is important to study the behavior of the material at such severe service conditions. Ti-modified 14Cr–15Ni austenitic stainless steel [2] has been chosen in cold worked condition as a fuel clad material for fast breeder nuclear reactor [3]. Cold work induces imperfections in lattice such as dislocations, which interact with point defects generated by neutron irradiation and thus material resists creep as well as void swelling [4]. Addition of Ti in the alloy enables the formation of fine titanium carbides on
dislocations during creep exposure, which improves the creep strength and especially void swelling resistance [5]. Hence, a better understanding of the creep behavior of such steel at various cold work conditions is required. The creep properties of cold worked Ti-modified 14Cr–15Ni austenitic stainless steel are compared with those of as received material and effect of cold work on creep properties along with microstructural changes are studied in this paper.

2. Experimental Work

Ti-modified 14Cr–15Ni austenitic stainless steel was obtained in mill annealed condition in the form of rolled plate with 13 mm thickness. The chemical composition of steel is given in Table 1. The plate was cut into bars of 120 x 50 x 13 mm along the rolling direction. These bars were solution annealed at 1333 K for 30 minutes. After the solution annealing of the alloy (CW0), 40% deformation was effected by cold rolling (CW4) at room temperature with the help of rolling mill (Buhler make). Creep specimens with gauge diameter of 5 mm and gauge length of 50 mm were then fabricated as per ASTM E139 standard. Creep tests were carried out on the annealed (CW0) as well as cold rolled samples (CW4) at 923 K with uniaxial creep testing machines (Star Testing make). The stress levels were in the rage of 175 – 225MPa.

For microstructural study, standard metallographic polishing was done followed by electrochemical etching at 6 V using 10% oxalic acid solution. Metallographic studies were carried out using (Olympus make) optical microscope and JEOL 6380 scanning electron microscope (SEM). Microhardness measurement was done with Mitutoyo microhardness tester at 0.3 kgf load with dwell time of 10 seconds. From each sample, average of 10 measurements was made. The X-ray diffraction (XRD) profiles were obtained using PAN alytical X’Pert Pro MPD diffractometer using CuKα radiations. The grain size was measured with the help of ImageJ software.

Table 1. Chemical composition of test material in wt%.

| Element | C   | Si  | Mn  | P   | S   | Cr  | Ni  |
|---------|-----|-----|-----|-----|-----|-----|-----|
| Wt%     | 0.041 | 0.745 | 2.491 | 0.023 | 0.002 | 14.091 | 15.523 |
| Element | Mo  | B   | N   | V   | Nb  | Ti  | Fe  |
| Wt%     | 2.214 | 0.005 | 0.0087 | 0.022 | 0.023 | 0.232 | bal |

3. Results & Discussion

3.1 Microstructure

Microstructure of the solution annealed 14Cr–15Ni austenitic stainless steel consists of equiaxed grains as shown in Fig. 1a. The initial grain size of the solution annealed steel was approximately 36µm. Cold work produced a banded structure as shown in scanning electron micrograph of 40 % cold worked (CW4) sample in Fig. 1c. Grains were elongated along the bands. Before creep testing, relatively less precipitation of carbide could be found in microstructure of CW4 sample as compared to that in exposed sample while precipitation was not observed in CW0.

From XRD profiles of CW0 and CW4 shown in Fig. 2, it can be clearly seen that, peaks have been shifted towards right and peaks broadening has occurred. It suggests that, lattice strain is accumulated on account of cold working. Dislocation density was multiplied during cold working of steel and dislocation motion was reduced due to less inter barrier spacing for dislocations, which contributed to better mechanical properties of the material [6].
Emergence of strong peak of (220) plane in XRD pattern of creep exposed CW4 sample indicates occurrence of recrystallization during creep. Such peak is not as strong in creep exposed CW0 samples.

**Figure 1.** Optical micrograph of (a) solution annealed sample, (b) 40% cold worked (CW4) sample and Scanning electron micrograph of (c) banded structure in CW4.
3.2 Hardness

Variations in microhardness of the CW0 and CW4 samples before and after creep testing are shown in Table 2. It can be observed that 40 % cold working doubled the initial hardness. This increased hardness was the manifestation of high strain energy stored in the material in the form of large number of dislocations. However, after creep exposure at 973 K, CW0 steel samples exhibited increased hardness at higher stress level, where the mechanical failure was more dominant as compared to metallurgical failure. Material experienced strain hardening due to high stress level and less creep exposure time. However, during comparatively long creep exposure of CW4 steel samples, the hardness was found to be decreased significantly for all stress levels. Comparing these two test conditions, the decrease in hardness in CW4 confirmed by XRD and microscopy indicates recovery and recrystallization during creep exposure.

CW0 samples showed no sign of recrystallization while strain hardening was observed. There was no appreciable change in hardness occurred as compared to CW4, wherein recrystallization effect has resulted in strain free grains leading to drastic reduction in hardness during creep exposure.

Table 2. Microhardness of CW0 and CW4 before and after creep test.

| Cold Work | Before Creep | After Creep |
|-----------|--------------|-------------|
|           |              | Stress      | Hardness (HV) |
| CW0       | 150          | 225         | 172           |
|           |              | 200         | 159           |
|           |              | 175         | 143           |
| CW4       | 355          | 225         | 240           |
|           |              | 200         | 231           |
|           |              | 175         | 212           |
3.3 Creep Properties

The stress vs creep rupture life of the CW0 and CW4 samples at 973 K is shown in Fig. 3. A linear relationship was found between stress and rupture life of both type of samples. Significant increase in creep life was found after cold working.

![Figure 3. Stress vs rupture life of CW0 and CW4](image)

Creep curves of the samples, CW0 and CW4 tested at 973 K under different stress levels 225, 200 and 175 MPa are compared in Fig. 4a, b and c, respectively. Cold worked sample (CW4) exhibited negligible primary creep deformation as compared to the mill annealed sample (CW0). CW4 exhibited longer secondary stage of creep deformation than the CW0. Parameters related to creep deformation and rupture of CW0 and CW4 are shown in Table 3. Minimum creep rate or steady state creep rate was found to be lesser in CW4 as compared to CW0, which also indicated the higher creep life of CW4 steel. Rupture life of the steel in the solution annealed condition (CW0) was lower than that of the cold worked condition (CW4) for a particular stress level.

Strain at rupture or rupture elongation was more in CW0 as compared to CW4, which indicated that CW0 exhibited higher ductility than CW4. It could be due to the formation of martensite during cold working in CW4, which resulted in the lower ductility during creep exposure [7].

| Cold work (%) | Temperature (°C) | Applied stress (MPa) | Rupture life (Hrs) | Minimum creep Rate (s⁻¹) | Rupture elongation (%) | Area reduction (%) |
|--------------|------------------|----------------------|--------------------|--------------------------|-----------------------|-------------------|
| CW0          | 700              | 225                  | 38                 | 1.5E-3                   | 7.8                   | 67                |
|              |                  | 200                  | 155                | 3.9E-4                   | 13.7                  | 74                |
|              |                  | 175                  | 378                | 9.2E-5                   | 27.1                  | 72                |
| CW4          | 700              | 225                  | 481                | 5.8E-5                   | 12.7                  | 59                |
|              |                  | 200                  | 1470               | 1.9E-5                   | 14.7                  | 68                |
|              |                  | 175                  | 2490               | 1E-5                     | 9.9                   | 63                |
Figure 4. Creep curves of CW0 and CW4 steel at (a) 225 MPa, (b) 200 MPa and (c) 175 MPa stress.

CW4 samples showed minimum creep rate with longer secondary region and lead to better creep strength. Percentage area reduction was also found to be on lower side in CW4 as compared to CW0 indicating reduced ductility. Lesser ductility in CW4 can be attributed to high hardness on account of high dislocation density and banded structure. However, percentage area reduction for CW4 samples was found to be close to those for CW0 samples. It can be inferred that, recrystallization phenomenon during creep has restored the ductility in heavily worked CW4 samples.

Optical micrographs of the creep tested CW0 and CW4 samples at 973 K at 200 MPa are shown in Fig. 5a and b, respectively. Extensive intra and inter-granular precipitations of carbides were observed after creep. After creep test, average grain size of CW0 steel samples was found to be 26 μm whereas CW4 samples showed a wide range of grain size from 10 to 105 μm as shown in figure. Due to prior cold work, the CW4 sample experienced recovery and recrystallization phenomena during creep testing, which led to the variation in grain size. Microstructure of CW4 sample revealed that, some
grains were precipitation free and bigger in size as compared to other grains. The micrographs of creep tested samples of CW4 revealed a well recovered sub-grain structure and recrystallized grains. Latha et al reported that after solution annealing primary Ti carbides re-precipitated during creep exposure which provides better creep strength [8].

Figure 5. Optical micrographs of ruptured samples (stress= 200 MPa) of (a) CW0 and (b) CW4

The austenitic stainless steel exhibits higher creep resistance, which comes from solid solution strengthening and carbide precipitation in the matrix. These precipitations predominantly occur near grain boundaries and reduce the dislocation movement. During creep exposure, precipitates coarsen and lead to the failure of the material [9]. Vijayanand et al also reported that, due to presence of titanium in the matrix, fine secondary titanium carbides precipitate preferentially in intra-granular dislocation network while M23C6 type carbides precipitates near grain boundaries [10]. These fine precipitates help to retard the dislocation movement in the matrix and decrease the recovery and recrystallization rate in cold worked structure, which result in better creep strength.

4. Conclusion
Based on the initial investigation of effect of cold work on creep behavior of 14Cr–15Ni austenitic stainless steel, following conclusions were derived:

1) Cold work induced dislocation network contributes to increased hardness as well as creep resistance in Ti-modified 14Cr–15Ni austenitic stainless steel as compared to that in mill annealed condition.
2) Post test microstructural analysis confirmed the occurrence of partial recrystallization during creep testing of cold worked Ti-modified 14Cr–15Ni austenitic stainless steel.

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