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Effect of cold-work on self-welding susceptibility of austenitic stainless steel (alloy D9) in high temperature flowing sodium

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ABSTRACT

Self-welding susceptibility of alloy D9 (15Cr–15Ni–2Mo titanium–modified austenitic stainless steel), used as wrapper in the fuel subassemblies of sodium cooled fast reactor, was studied in flowing sodium. Specimens were tested at 823 K in annealed and in 20% cold-worked condition up to a maximum contact stress of 24.5 MPa and maximum duration of 9 months. The results showed that the annealed alloy D9 showed good resistance to self-welding in all the tests. But 20% cold-worked alloy D9 got self-welded in all the tests except in the test carried out for 3 months duration indicating that tests conducted at high contact stresses and long duration reduce the resistance of the steel to self-weld. Microstructural changes observed in the cold-worked alloy D9 at the location of contact between the mating surfaces indicate dynamic recovery resulting from high contact stress and temperature facilitating self-weld.

1. Introduction

In sodium cooled fast reactor, core contains fuel subassemblies where the fuel pins are packed in a hexagonal wrapper. Minimum contact area between the fuel subassemblies is set by providing wrapper pads on each of the six faces of the hexagonal wrapper. For India's Prototype Fast Breeder Reactor (PFBR), which is under construction, 20% cold-worked alloy D9 is the material chosen for hexagonal wrapper of fuel subassemblies and wrapper pads. Cold-work is given to impart improved resistance to void swelling for the alloy D9. During the operation of the reactor, the maximum contact stress to which some of the wrapper pads are subjected to is estimated as 3.3 MPa at 823 K. It is important that high operating temperature, contact stress and fairly long resident time (24 months) of the subassemblies inside the reactor do not lead to self-welding of the subassemblies at the wrapper pads. In the event of self-welding of the wrapper pads, additional force has to be applied to separate the self-welded subassemblies during fuel handling operation.

Self-welding is essentially a diffusion bonding phenomenon that occurs when two virgin metallic surfaces are pressed against each other for a particular duration at high temperature. During this process, deformation of the surfaces in contact takes place due to loading and during the subsequent recovery and recrystallisation that follow, diffusion of atoms takes place across the contact interface resulting in self-welding of the mating surfaces [1]. Liquid sodium in inert environment removes the oxide films that would normally be present on the metallic surfaces, and thus facilitates self-welding.

Limited literature is only available on self-welding studies on stainless steel in sodium. Few sodium experiments were reported in 304, 316 and 321 stainless steel. Relations between the breakaway shear force with contact pressure, contact temperature and surface finish were established [2,3]. The self-welding coefficient (W), defined as the shear stress for breakaway per unit contact stress, is related to square root of duration (t) of the self-welding test [4]. Agastini has studied self-welding in 20% cold-worked AISI 316 stainless steel [5]. Recently self-welding studies on annealed and cold-worked alloy D9, and 316LN austenitic stainless steel [6,7] were reported by our team.

As 20% cold-worked alloy D9 exhibited susceptibility to self-welding, it is important to understand the mechanism of self-welding of this alloy in the cold-worked condition and also to ensure that self-welding does not take place in the hexagonal wrapper pads of PFBR during the residence time of the reactor. Hence, the present study was conducted.

2. Test facility for self-welding

The test set-up for evaluating susceptibility to self-welding is shown in Figs. 1 and 2. It is provided with a rod, outer tube, disc spring, nut, load cell and provision to hold the specimens at bottom. The specimens for studying self-welding susceptibility are hollow cylinders of 21.4 mm OD, 15.8 mm ID and 15 mm height.

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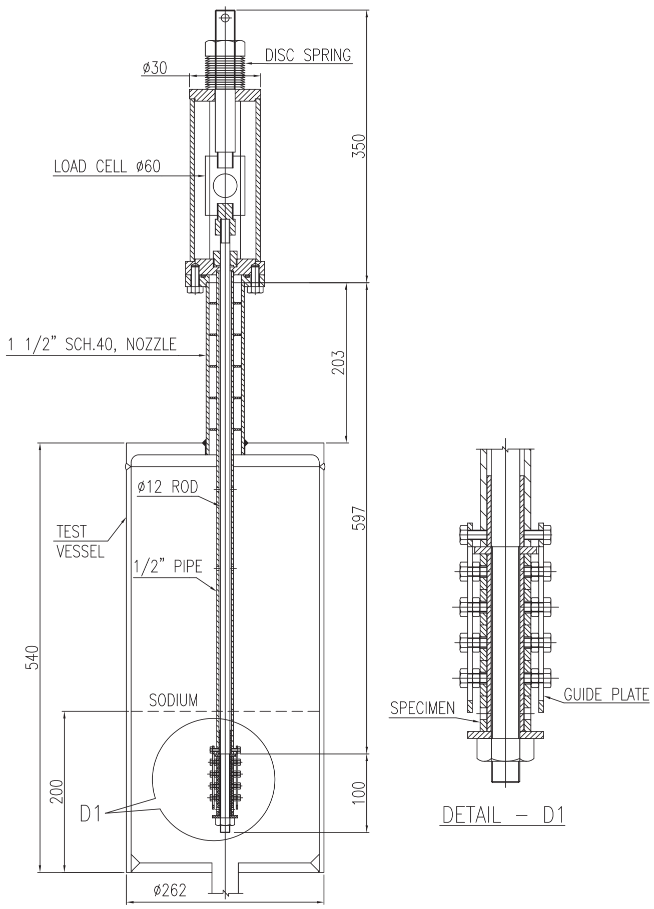


Fig. 1. The sodium test vessel.

The top and bottom surfaces of the hollow cylindrical specimens are the mating surfaces for the test. In some specimens the top contact area is reduced by providing a step to increase the contact stress. Fig. 2a shows one of the specimens with a step and Fig. 2b shows the assembly of specimens. The contact pressure is applied by tightening disc springs at the top. Load cell is connected to the mechanism to measure the applied load. The surface roughness of the contact area is maintained below 0.5 μm. The set-up is introduced into a vessel and sodium is filled to immerse the specimens. Test vessel is connected to a sodium loop with a purification facility using cold trap. Reactor grade purity (99.95% pure) is maintained for flowing sodium with oxygen concentration less than 2 ppm. The sodium temperature is maintained at 823 K with sodium flow rate of 2l/min and the sodium loop is operated continuously till experiment is completed. After the completion of the test, specimens that are self-welded were separated using a set-up as shown in Fig. 3 which consists of mechanical vice, bolt nut, disc springs and load cell with which the breakaway force required to separate them can be measured [7].

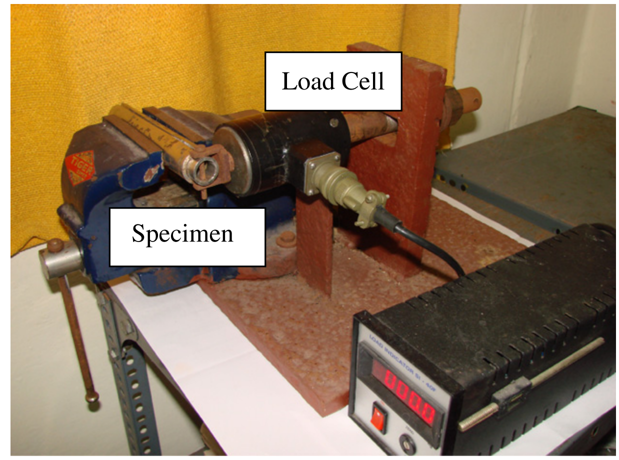
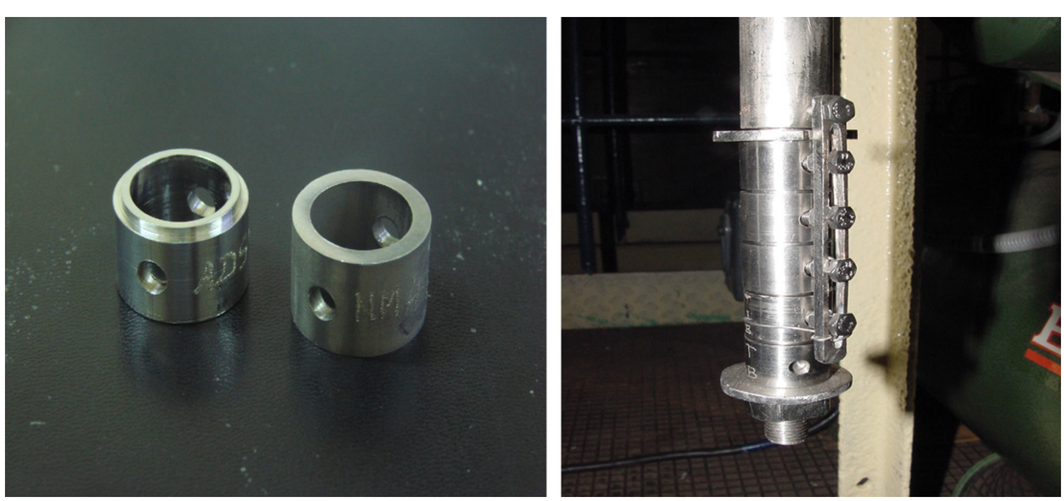


Fig. 3. A set-up for measuring the breakaway force [7].

3. Experimental procedure

Composition of alloy D9 is shown in Table 1. Tests were conducted both using annealed alloy D9 and 20 % cold-worked alloy D9. Annealing of alloy D9 is by heating to 1353 K for 1 h and cooling to room temperature. For imparting cold-work, alloy D9 rod is pulled in tensile testing machine at a constant rate at room temperature till the area of cross section of the rod is reduced by 20%. Specimens are assembled in the test set-up and introduced into the vessel with an initial gap of 1–2 mm between the specimens. Sodium flows in the test vessel and specimens are immersed



(a) Specimens (b) Assembly of Specimens

Fig. 2. Specimen assembly for insertion into sodium vessel.

Table 1
Chemical compositions of alloy D9 (in wt.%).

Material	Ni	Cr	Mo	Mn	Si	Ti	C	P	S	N	Fe
Alloy D9	15.07	15.05	2.25	1.51	0.51	0.32	0.05	0.01	0.0025	66 ppm	Balance

Table 2
Results of self-welding tests in sodium at 823 K.

Material combination	Dwell Time *t* (months)	Contact force (N)	Contact area (mm^2)	Contact pressure (MPa)	Self-welding behaviour	No. of pairs tested	No. of pairs self-welded	Break-away shear force (N)	Self-welding coefficient *W* (break- away/contact force)	$t^{1/2}$
20%CW D9 vs. 20%CW D9	3 (2160 h)	1566	166.5	9.4	No self-welding	2	Nil			46.4
20%CW D9 vs. 20%CW D9	4.5 (3240 h)	2000	81.6	24.5	Self-welded	3	1	182	0.1	56.9
20%CW D9 vs. 20%CW D9	6 (4320 h)	1566	166.5	9.4	Self-welded	2	1	202	0.13	65.7
20%CW D9 vs. 20%CW D9	9 (6480 h)	2000	166.5	12	Self-welded	4	2	330, 252	0.16, 0.13	80.5
Annealed D9 vs. annealed D9	3 (2160 h)	1566	166.5	9.4	No self-welding	1	Nil		0	46.4
Annealed D9 vs. annealed D9	4.5 (3240 h)	2000	81.6	24.5	No self-welding	3	Nil		0	56.9
Annealed D9 vs. annealed D9	6 (4320 h)	1566	166.5	9.4	No self-welding	1	Nil		0	65.7

in the flowing sodium. The sodium temperature is maintained at 823 K. Pure sodium reacts with the mating surfaces and removes the oxide layer and pure metal surface is exposed. Subsequently the specimens are loaded by tightening the disc spring. The experiment is carried out for duration of 3–9 months. After the experiment the sodium is drained, the test set-up was cooled to room temperature and the specimens are removed from the sodium loop for examination. For specimens that are self-welded the shear force to separate the specimens was measured. The experimental condition and the results are shown in Table 2.

4. Results and discussions

From the results shown in Table 2, it is clear that none of the annealed alloy D9 specimen pairs got self-welded. In contrast, self-welding was observed in the cold-worked specimens for the tests conducted for durations of 4.5, 6 and 9 months, though for tests conducted for durations of 3 months, there was no self-welding. The breakaway force required to separate the specimen was found to increase with increase in duration of the test. The contact stress in the experiment is estimated assuming 100% contact between two mating surfaces, however the actual contact area will be less as the specimens will be in contact only along asperities of the mating surface of specimen. Hence the factor self-welding coefficient is defined.

Though the specimens of same material combinations were tested under same experimental conditions, all the pairs did not self-weld equally. This is due to variations in the actual contact area at the asperities of the mating surfaces of the specimens during loading [7]. The pair with relatively minimum contact area at the asperities of the mating surfaces experiences high contact stress and gets self-welded. Other pairs with weak self-weld break up during cleaning and removal from experimental set-up. Hence the pairs with strong bonding and separated by force were reported as self-welded specimen.

Variation of self-welding coefficient (*W*) against square root of time of testing ($t^{1/2}$) for annealed D9 and 20% cold-worked alloy D9 are shown in Fig. 4. Linear relationship established between W and $t^{1/2}$ with test results of duration 6 and 9 months [7] seems

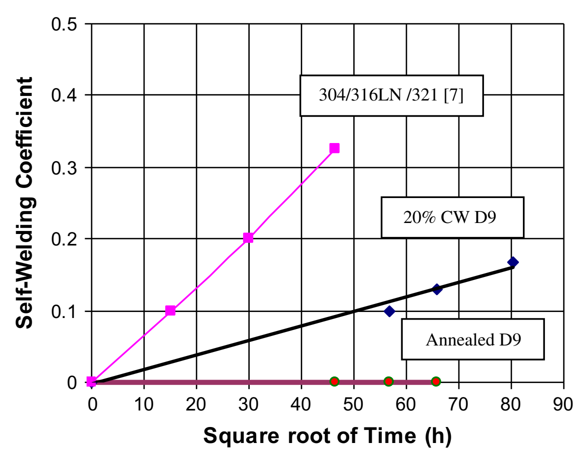


Fig. 4. Self-welding susceptibility of annealed D9 and 20% cold-worked D9 in sodium at 823 K.

to hold even for test of 4.5 month duration carried out at higher contact stress. Since annealed alloy D9 did not show self-welding, the corresponding variation for these specimens will be as shown in Fig. 4. The test results of 304 and 321 stainless steel of Yukota [4] and 316LN obtained from our earlier study [7] fall in same line as shown in Fig. 4. From the figure it is clear that annealed D9 is least susceptible for self-welding, followed by cold-worked alloy D9 and then 304/321/316LN stainless steel.

Specimen surfaces were examined using scanning electron microscopy (SEM) for surface damage after testing. The micrographs, shown in Fig. 5, reveal that there is no surface damage in the annealed specimen even at higher contact pressure of 24.5 MPa for 4.5 months. Machine marks present on the surface of the specimen before start of the test can be easily seen. In contrast, for the 20% cold-worked alloy D9 specimens that were self-welded, the surface damage is clear as shown in Figs. 6 and 7. Entire surface was not self-welded; it occurred predominantly at the asperities present along the mating surfaces.

Microstructural investigations and hardness measurements were carried out on annealed and cold-worked alloy D9 specimens

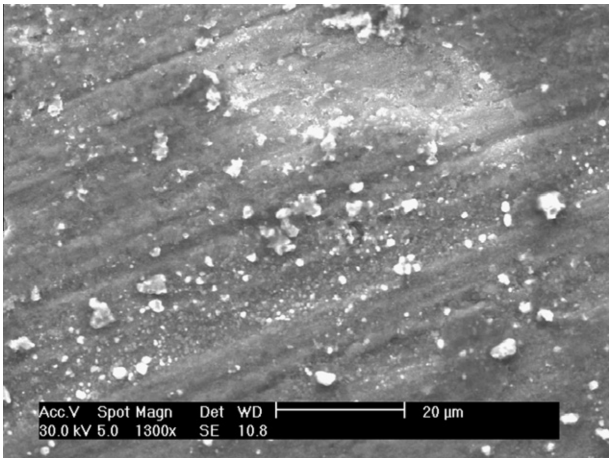


Fig. 5. Annealed alloy D9 after 4.5 months and contact pressure 24.5 MPa in flowing sodium at 823 K.

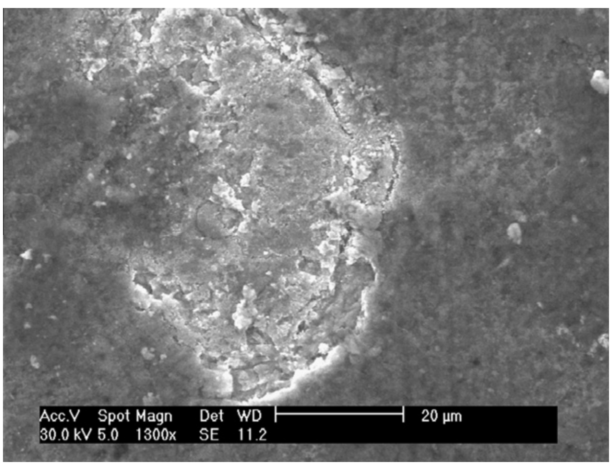


Fig. 6. 20% Cold-worked alloy D9 after 4.5 months and contact pressure 24.5 MPa in flowing sodium at 823 K.

in order to find out the possible causes for high susceptibility of cold-worked alloy D9 specimens to self-welding. Microstructures of annealed and 20% cold-worked alloy D9 before testing are compared in Fig. 8 [6]. In the annealed specimens, annealing twins are observed and in the cold-worked specimen twin density is much higher than the other and these twins are generated due to cold-work.

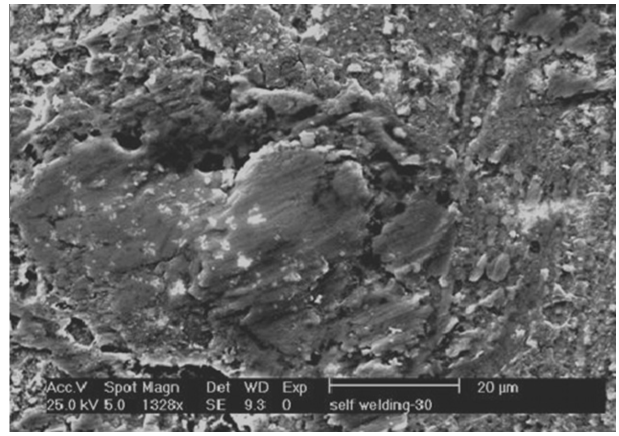


Fig. 7. 20% Cold-worked alloy D9 after 6 months and contact pressure 9.4 MPa in flowing sodium at 823 K.

Microstructural examinations were carried out at different locations of the specimen after self-welding test. Fig. 9 shows the locations in the 20% cold-worked specimen that was self-welded after a test of 4.5 months duration at 24.5 MPa contact stress. AB is the thickness of the cylindrical specimen. Microstructures at points corresponding to point C and E in Fig. 9 are shown in Fig. 10a and b respectively. Location C experienced direct loading during testing while the location E was not under direct loading. The cold-worked microstructure at location C has undergone recovery than the microstructure at location E. In annealed alloy D9 microstructure examination after the experiment showed that the twin boundaries are reduced as shown in Fig. 11.

As, good difference in microstructures was noticed at locations C and E, hardness profile along the length of the specimens was taken at locations C, D and E shown in Fig. 9 and the results are shown in Table 3. Hardness values along the length at locations C and D, which were under direct loading during test are lower than that at location E which was not under direct load during testing. This is again an indication of recovery of cold-worked microstructure at locations C and D under applied load. This hardness reduction is not caused by ageing due to exposure to high temperature as it is observed only at location C and D and not at E.

It is earlier reported that during testing, specimens pick up carbon from the flowing sodium and there is slight increase in carbon content and hardness at the specimen surface [6,7]. Decoration of austenite grain boundaries with carbides near the specimen surface has also been established. Hence, microstructure at locations C and E were examined under SEM to find out whether there is

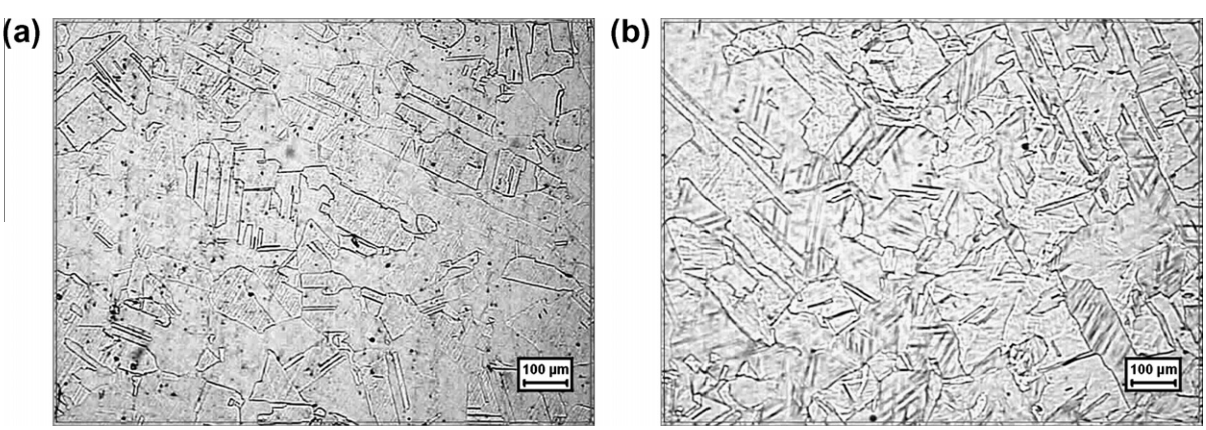


Fig. 8. Microstructure of (a) annealed D9 and (b) 20% cold-worked alloy D9 specimens before self-welding susceptibility studies [6].

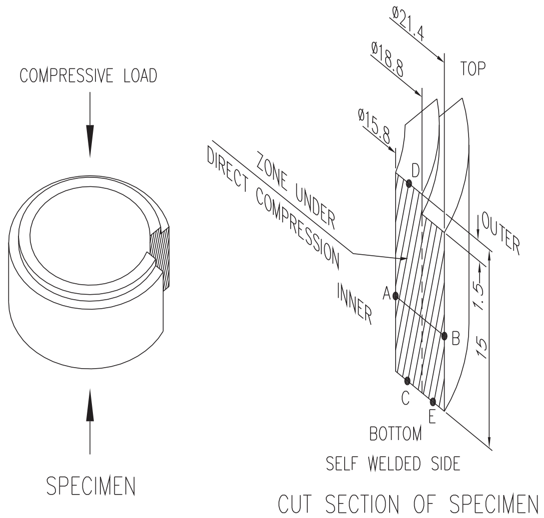
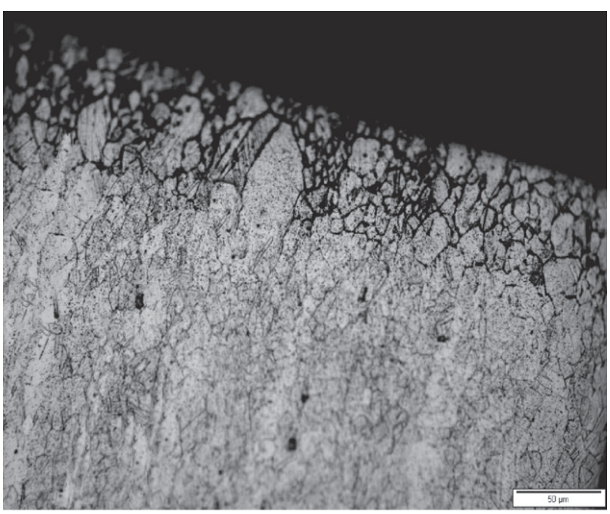
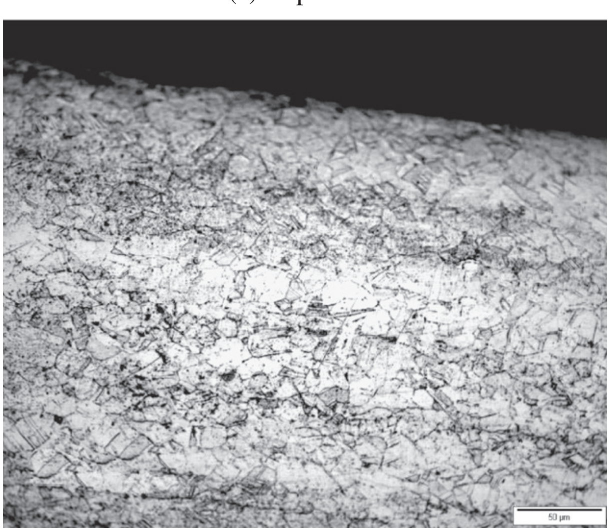


Fig. 9. Cut section of self-welded 20% cold-worked specimen for metallographic analysis.



(a) At point C



(b) At point E

Fig. 10. Microstructure of 20% cold-worked specimen after 4.5 months in 823 K flowing sodium at 24.5 MPa contact pressure.

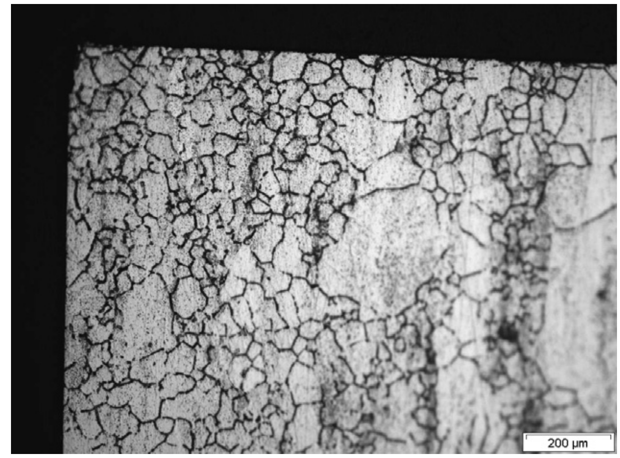


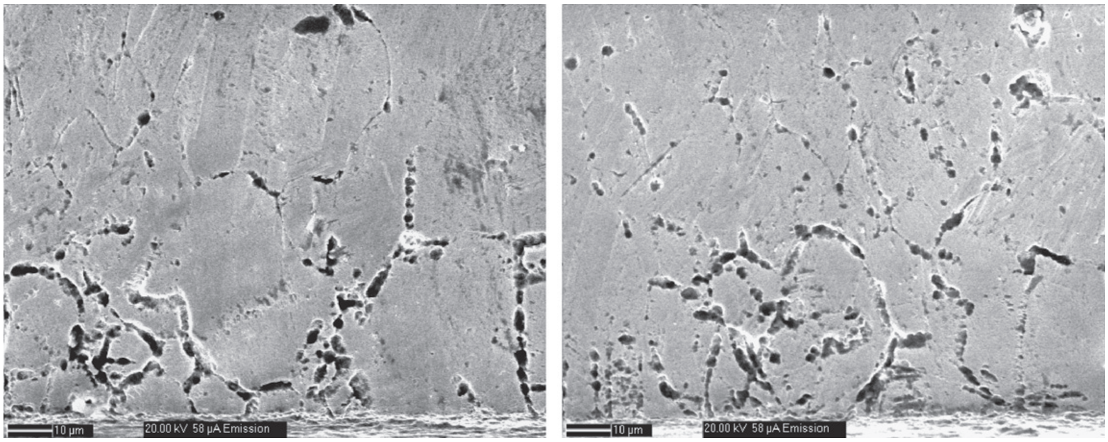
Fig. 11. Microstructure of annealed specimen after 4.5 months in 823 K flowing sodium at 24.5 MPa contact pressure.

Table 3
Hardness of 20% cold-worked alloy D9 after sodium testing for 4.5 months at 823 K in flowing sodium at 24.5 MPa contact pressure.

Depth (mm)	Hardness (VHN) of self-welded sample		
	At point C recrystallized side	At point E non-recrystallized side	At point D recrystallized side
0.2	234	291	210
0.4	217	265	214
0.6	238	278	207
0.8	223	259	187
1	204	242	196

any difference in the carbon pick up and subsequent difference in the carbide distribution at these locations. Fig. 12a and b show SEM micrographs very close to the specimen surface at locations C and E of Fig. 9 respectively. There is no difference in the carbide distribution observed at both these locations. Hence, observed difference in the microstructure and hardness at these two locations of the 20% cold-worked alloy D9 could not be attributed to difference in carbon pick up from liquid sodium.

From the results presented above, it is clear that recovery and recrystallisation of the microstructure has taken place in the cold-worked alloy D9 specimen that were self-welded after testing for a duration 4.5 months at a stress level of 24.5 MPa. The fact that recrystallisation was not observed at location of the specimen that was not under direct loading indicate that dynamic recovery and recrystallisation is assisted by stress. As recovery and recrystallisation is also assisted by diffusion, it is clear that self-welding is facilitated by the recovery and recrystallisation of the work hardened microstructure of the cold-worked alloy under stress at high temperature for prolonged duration. It is true that the microstructural examination did not show significant recrystallisation for cold-worked specimens that were self-welded in tests conducted at longer durations of six and nine months with lower stresses [7]. This is due to slow kinetics of recovery and recrystallisation at lower stress levels, and as dynamic recovery is assisted by stress, higher the stress faster is the recovery. In this context, it is appropriate to note that recovery of the cold-worked alloy D9 during high temperature ageing has been studied in details and it has been shown that during ageing at 823 K, kinetics of recovery is very slow and hardness measurement after ageing does not reveal any significant reduction [8]. Hence, dynamic recovery of cold-worked microstructure assisted by high contact stress and temperature is the cause for higher self-welding susceptibility in cold-worked alloy D9 than in annealed alloy D9.



(a) SEM at point C which is under direct load (b) SEM at point E which is not under load

Fig. 12. SEM micrograph of 20%cold-worked specimen after 4.5 months in 823 K flowing sodium at 24.5 MPa contact pressure.

As already stated, it is important to ensure that self-welding between the subassemblies made of 20% cold-worked alloy D9 does not take place during reactor operation. The results from the present study confirm that the cold-worked alloy D9 is susceptible for self-welding, and the duration for self-welding to take place depends on contact stress also. Instead of testing with contact stress of 3.3 MPa for 24 months the contact stress required for the accelerated test of 3 months is determined using the equation $W = Kt^{1/2}$.

$$W_{24} = Kt_{24}^{1/2} \quad (1)$$

$$W_3 = Kt_3^{1/2} \quad (2)$$

where W_{24} is the self-welding coefficient for 24 months (t_{24}), and W_3 is the self-welding coefficient for 3 months (t_3) and K is the constant.

Dividing Eqs. (1) and (2), the ratio of W_{24}–W_3 is 2.8. Assuming that self-welding take place and breakaway shear force would be same for both durations, the contact stress for three months is estimated as (3.3×2.8) 9.4 MPa. The 20% cold-worked specimen tested in sodium for three months with a contact stress of 9.4 MPa did not self-weld. By the relation shown in Fig. 4 for 20% cold-worked alloy D9, only a weak bonding would have taken place in the mating surfaces of the specimens for the contact stress of 9.4 MPa. Hence, it can be concluded that self-welding is unlikely to take place in the hexagonal wrapper pads made of 20% cold-worked alloy D9 under PFBR operating conditions.

5. Conclusions

Major conclusions from the present study are the following:

- Cold-working of the alloy D9 makes it susceptible to self-welding due to dynamic recovery of cold-worked microstructure under high stress and temperature. However, self-welding of hexagonal wrapper pads of fuel subassembly of PFBR made of 20% cold-worked alloy D9 is unlikely to take place.
- The annealed alloy D9 is more resistant to self-welding than 20% cold-worked alloy D9.

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