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Experience with irradiation of LiSoR test section no. 3

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Abstract

LiSoR (liquid metal–solid metal reaction) loop is a unique facility which was designed to investigate simultaneously the influence of flowing lead bismuth eutectic (LBE), static stress and irradiation by protons onto steel that might be used as structural material in a future ADS (accelerator driven system) reactor. LiSoR is worldwide the first LBE loop that is under operation while it is irradiated at the same time.

Up to now five LiSoR test sections have been irradiated. In this paper the experience on LiSoR no. 3 is presented including irradiation experiment and dissembling of the test section and EDM wire cutting of the samples. Additionally the results obtained by SEM and EDX analyses on LiSoR specimen and on beam window are shown whereby the most interesting outcome is the finding of an oxide layer, which was clearly identified by X-ray mapping. This layer is located directly in the irradiated area having a total thickness less than 1 μm. Additionally solidified LBE was detected on some areas of the samples but no wetting of the steel or penetration of LBE into the steel matrix was observed. No enrichment or dissolution of steel elements such as Fe and Cr could be detected on the surface near area. The steel surface is still smooth without any indications of corrosion attack.

1. Introduction

Lead–bismuth eutectic (LBE) alloy is beside lead one of the prime candidates to be applied as target material for a future accelerator driven system (ADS) reactor. Meanwhile there exist several experimental facilities within Europe, Japan and USA that are used to investigate the material behaviour of different kind of steels in lead and mainly LBE.

A broad set of different parameters like flow rates, temperature ranges and oxygen content in LBE is chosen to expose steel specimens. Thanks to the strong effort undertaken during the past in this field liquid metal corrosion is relatively well understood and thus special features exist helping to suppress it (Fazio et al., 2001; Glasbrenner et al., 2001; Barbier and Rusanov, 2001; Anonymous, 2003). The other harmful reaction is the so-called liquid metal embrittlement (LME) and cannot be predict easily. LME can lead to a severe and very fast unusually brittle intergranular failure of ductile materials (Nicholas and Old, 1979; Kamdar, 1983).

The behaviour of structural material under irradiation in the presence of LBE and static stress and the influences on LME is another great uncertainty, which needs to be investigated. Therefore the LiSoR (liquid–solid reaction under irradiation) experiment was launched to investigate the effects of liquid metal embrittlement and corrosion directly during irradiation (Kirchner et al., 2003).

LiSoR setup is basically a LBE loop with a test section irradiated with 72 MeV protons whereby so far five test sections including T91 specimens have been irradiated in the presence of flowing LBE (Glasbrenner et al., 2002, 2003, 2005b, 2005a). During these irradiation runs various experiences were gathered, some of them very constructive, other rather disappointing. At the end we gained experiences, although disappointing at the first glance, turned out to be very valuable for finding ground in a complete unexploited field.

The LiSoR loop with test section no. 3 operated for 264 h under proton irradiation with a beam current of 15.5 μA and a beam energy of 72 MeV (Glasbrenner et al., 2003). The maximum temperature in the inner wall of the target window was calculated to be around 342 °C under these specific conditions. Due to an erroneous signal of a leak foil in the beam line, the irradiation experiment had to be stopped ahead of schedule. SEM and EDX analyses of materials exposed to LiSoR loop during irradiation of LiSoR test section no. 3 are now completed and are reported in this paper.

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2. Experimental

2.1. Test facility LiSoR

The loop plus test section was designed by Subatech, France; the test section in total was manufactured by SOTEREM, France; the liquid metal pump and flow meter was constructed at IPUL, Latvia; assembling, cabling, isolating, filling with LBE and commissioning was performed at PSI, Switzerland. The loop was fabricated of austenitic stainless steel 316L whereas in the test section ferritic steel T91 was used. LiSoR installed in the IP1 bunker is connected to a beam line from the Philips PSI accelerator. More details of the loop are described in (Kirchner et al., 2003; Cadiou, 2002; Dementjev et al., 2001).

About 18 l of LBE were filled into the melting vessel of LiSoR while during operation about 15 l of LBE are pump around in the loop. The eutectic Pb–55.5Bi (44.8 wt.% Pb and 55.2 wt.% Bi) alloy used was supplied by Impag AG (Switzerland). The chemical analysis of LBE performed at PSI before irradiation has given a few ppm of impurities: Ag 11.4, Fe 0.78, Ni 0.42, Sn 13.3, Cd 2.89, Al 0.3, Cu 9.8, Zn 0.2.

2.2. Test specimen

LiSoR specimen was made of DIN 1.4903 steel 9Cr1MoVNb named T91 supplied by the company Ugine, France. The material is from the same heat as that used in the EU 5th Framework program SPIRE. The 15 mm thick steel plate was delivered in standard condition, i.e. the material was normalised at 1040 °C for 60 min and tempered at 760 °C for 60 min. The composition in wt.% is 8.63 Cr, 0.23 Ni, 0.95 Mo, 0.31 Si, 0.43 Mn, 0.1 C, 0.21 V, 0.02 P, 0.09 Nb and with the balance Fe.

The specimen was prepared in the dimensions shown in Fig. 1. The orientation of the specimen in the plate is parallel to the rolling direction. The gauge area was mechanically polished and finished with polishing papers of no. 2000. The specimen was degreased with ethanol and acetone before installation into the LiSoR test section.

2.3. Test section

The ovoid cross section of the tube was chosen to provide a flat irradiation area comparable to the test specimen as well as to minimize mechanical stress due to differential pressure between the lead bismuth loop and the beam line channel which is under vacuum (i.e. 0.25 MPa of total pressure between the two gives rise to 30 MPa of mechanical stress). The profile and cross section of the tube including the specimen and liquid LBE are shown in Fig. 2. The tube of the test section is fabricated of standard heat-treated ferritic compact T91steel block; the raw material was purchased from Creusot Loire Industrie (France) and has a composition in wt.% of 8.26 Cr, 0.13 Ni, 0.95 Mo, 0.43 Si, 0.38 Mn, 0.1 C, 0.2 V, 0.017 P, 0.065 Nb and with Fe in balance.

The outer surface of the tube was milled; the inside of the tube cut by electron discharge was mechanically reworked in a two step process by hand to obtain a smooth and crack-free inner surface by company Brehm, Switzerland. At first an abrasive block was used having a grain size of 240–320 to remove the "damaged" layer produced during EDM wire cutting. The second step was polishing with diamond paste of 16 μm. The polishing procedure lasted about 5 h and should result in a shiny surface after metal removal of about 40 and 50 μm. A visible inspection was not possible due to the complex geometry of the test section and the difficulties to inspect micro cracks.

2.4. Operational conditions

The operation of the loop is set to run completely automatically. A control unit monitors different kind of signals continuously. As long as all signals response well the beam is provided from the accelerator. The flow rate of the pump is up to 0.3 l/s, which corresponds to a flow velocity of about 1 m/s in the test section. The LBE temperature in the test section was set to 300 °C before irradiation, which was measured directly with a thermocouple immersed in the melt. As soon as irradiation was started the temperature increased to around 320 °C. The proton beam generated by the Philips cyclotron at PSI did not impinge

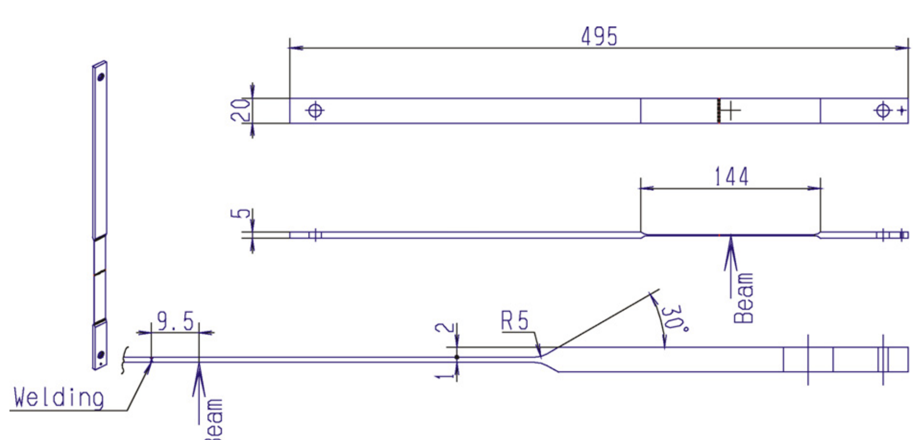

Fig. 1. Nominal dimensions of the welded specimen.