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Liquid metal embrittlement of silicon enriched steel for nuclear applications

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ABSTRACT

Lead–bismuth eutectic (LBE) liquid metal is considered to act as the coolant and as spallation neutron source for a future accelerator driven system (ADS). In addition to the highly corrosive effect of the liquid lead–bismuth eutectic, the liquid metal may also affect the mechanical integrity of the structural materials by a phenomenon called liquid metal embrittlement. Thus, although the addition of silicon to ferritic–martensitic steels has been found to strongly increase the material's resistance to corrosion in liquid lead–bismuth environment, their mechanical properties can be strongly affected by the liquid metal environment. This paper discusses the mechanical properties of an experimental silicon enriched high Cr/high creep resistant bainitic steel when in contact with LBE. The steel's mechanical properties were assessed by tensile testing as function of temperature in both liquid lead–bismuth eutectic and in an argon and hydrogen gas environment. It was found that the silicon enriched bainitic steel is very prone to liquid metal embrittlement between 300 and 375 °C with the transition in mechanical behaviour from ductile to predominantly brittle between 200 and 300 °C thus defining its the so-called ductility trough.

1. Introduction

Accelerator driven systems (ADS) and/or heavy liquid metal cooled fast reactors (LFR) are foreseen to play a dominant role in the back end of several of the advanced fuel cycle strategies [1,2]. Although it would be desirable for energy conversion efficiency to use pure lead at high temperatures as a coolant and a spallation source for these nuclear systems, the first experimental systems will most likely be using liquid lead–bismuth eutectic which allows lower operating temperatures [3]. These lower operating temperatures, which would be in the range of 250–550 °C, could allow the use of commercially available high strength, creep resistant steels. However, compatibility of the candidate structural materials and cladding with the liquid lead or LBE is known to be one of the critical issues to allow development of the ADS and LFR.

Previous Russian studies [4,5] pointed out the importance of oxygen control in liquid lead and LBE coolants because the corrosion would be dominated by dissolution of alloy components (liquid metal corrosion) at low oxygen concentrations and by oxidation at middle and high oxygen concentrations. Using active oxygen control, the corrosion in LBE was mitigated for a 12 wt% Cr martensitic steel with Si addition developed in Russia [6]. Subsequent studies confirmed that addition of Si significantly improves a material's corrosion resistance in contact with LBE [7–10].

However, apart from degradation of the structural steels by liquid metal corrosion, these materials need to be examined for liquid metal embrittlement susceptibility as well. Generally, when solid metals are exposed to liquid metals and stress is applied, they may undergo abrupt brittle failure known as liquid metal embrittlement (LME). It is characterized by a premature brittle failure of an otherwise ductile material when placed in contact with specific liquid metal for the material under stress [12]. LME is of prime interest because of the risk of damage wherever the handling of components that have been in contact with liquid metals is required. The phenomenon depends on many parameters like metallurgical state, hardness, composition, temperature, strain rate, and stress [11–15]. Temperature and strain rate are of extreme importance and will directly affect the severity of the liquid metal embrittlement of a given liquid metal/solid metal couple. In many of the reported severe cases of LME, the latter is most severe at temperatures close to the melting point of the coolant metal. At a certain higher temperature, the phenomenon of LME disappears, so forming a temperature interval within which the material is susceptible to LME, known as the ductility trough [16]. The extent of this interval of temperatures where the strain to rupture is largely reduced is constant for a given strain rate and its high temperature end is reported to decrease with decreasing strain

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rate [16]. At a sufficiently low strain rate the occurrence of LME is reported to disappear [16]. In any case, when the test conditions are inside the ductility trough for a given solid metal/liquid metal couple, the tensile behaviour of the solid material can be significantly changed under influence of the liquid metal. For the commercially available ferritic–martensitic steel T91, the ductility trough was found to be between 300 and 450 °C [17]. However, in sharp contrast to what was mentioned above, the ferritic–martensitic steel T91 seems to embrittle at low strain rate and recovers its ductility when the strain rate is increased [18]. Furthermore, the lower temperature limit of the ductility trough of T91 is found to be considerably higher than the melting point of the LBE (T_m = 123 °C) [17].

This paper discusses the tensile behaviour of a bainitic silicon enriched steel when tested in contact with liquid lead–bismuth eutectic environment as function of temperature while applying a constant strain rate of $5 \times 10^{-6}\ s^{-1}$. To assess the effect of the liquid metal environment, comparison is made with similar tests on the same material performed in an argon and hydrogen gas mixture.

2. Experimental

All tests were carried out using Liquid Metal Embrittlement Testing Station 1 (LIMETS 1) which was especially designed for testing in liquid lead–bismuth eutectic environment. The machine is based on the three vessel concept, e.g. melting tank, dump tank and autoclave. The LBE material was delivered by Hetzel Metalle GmbH, Germany with a composition of 55.2 wt% Bi and 44.8 wt% Pb, 2 mg/g Cr and less than 1 mg/g Ni. The ingots were molten in the melting tank and transferred to the dump tank in the liquid state while leaving the slag layer in the melting tank thus avoiding contamination of the system. Inside the dump tank the LBE was stored and preconditioned before every test. The preconditioning was performed by bubbling argon gas or a mixture of 5% hydrogen and 95% argon through the liquid metal at a well-defined temperature. The tensile tests were performed using a 10 kN machine. The samples were tested in the autoclave which was filled with 2 l of preconditioned liquid lead–bismuth eutectic after evacuating to a primary vacuum and filling with a hydrogen and argon gas mixture. This design allows achieving clean LBE with a predefined and controlled oxygen concentration. During testing the oxygen control is maintained by continuous bubbling of the argon and hydrogen gas mixture.

Small size cylindrical tensile samples were used with a total length of 24 mm, a gauge length of 12 mm and a diameter of 2.4 mm. These samples were manufactured in L orientation (rolling direction) with a lathe machine and were subsequently mechanically polished up to a 4000 grid paper. Prior to testing the samples were degreased by ultrasonic cleaning in methanol and dried in air. The samples were measured using a digital profile projector (type Mutitoyo PJ300).

The chemical composition of the experimental elevated Si steel called S2439 through out this paper is given in Table 1. The material was supplied by Los Alamos National Laboratory, Los Alamos, NM, USA, and received a normalisation heat treatment at 1050 °C for 60 min, followed by air cooling and a subsequent tempering treatment at 760 °C for 60 min, followed by air cooling. This heat treatment was based on the normalisation and tempering heat treatment of HT-9, a 12 wt% Cr ferritic–martensitic commercial steel with a similar chemical composition to S2439 but having only about 0.4 wt% Si. It was shown by Wilcox et al. [19] that these types of steel show the lowest ductile to brittle transition temperature (DBTT) when given a high temperature normalisation treatment and a high temperature tempering treatment. The resulting microstructure of the elevated silicon steel S2439 is bainitic as can be seen in the optical microscopy image of Fig. 1. To obtain this microstructural image, the sample was first mechanically polished up to 1 μm paper and subsequently etched using Villella's reagent.

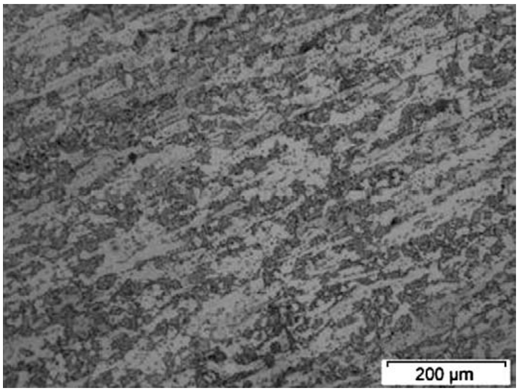

Fig. 1. Bainitic microstructure of the experimental elevated Si steel S2439.

The Vickers hardness of the S2439 steel was measured with a Wolpert DIATESTOR 2n using 5 kgf at 6 points. The obtained average Vickers hardness was 261 H_V. Furthermore, the steel's DBTT was determined using subsize KLST specimens. The material had a DBTT of 93 °C with an upper shelf energy of 3.6 J. The measured DBTT is considerably higher and the upper shelf energy is lower than this of HT-9 steel. This effect is caused by the addition of silicon. According to Harrelson et al. [20] addition of silicon does not adversely affect the DBTT of high Cr steels when the Si concentration remains below 0.4 wt%. When the silicon concentration is increased above 0.4 wt% however, the addition of Si has a detrimental effect on the impact properties of the steel. This was confirmed by Roy et al. [21] who examined the effect of silicon addition on the impact properties of ferritic–martensitic steel T91. In this study however, all tests are performed in the ductile temperature region, well above the DBTT of the S2439 steel.

Tensile tests were performed to assess the susceptibility of the S2439 steel to liquid LBE embrittlement. The effect of temperature was characterized by testing at temperatures between 150 °C and 500 °C at a constant strain rate of $5 \times 10^{-6}\ s^{-1}$. The influence of the liquid metal on the mechanical properties was determined by comparison with tensile tests performed using the same machine but in controlled gas environment.

After testing in liquid LBE, the samples were cleaned at room temperature in a chemical solution of hydrogen peroxide, ethanol and acetic acid in a 1:1:1 concentration ratio. This solution removes the solidified LBE without affecting the underlying base metal.

3. Results and discussion

The elastic and plastic flow behaviour of the Si enriched bainitic steel S2439 were found to be similar up to the point of fracture

Table 1
Chemical composition of experimental Si enriched steel S2439 (wt%).

	C	N	Al	Si	P	S	V	Cr	Mn	Ni	Mo	W
S2439	0.19	0.003	0.042	2.75	0.004	0.004	0.31	11.58	0.37	0.48	1.03	0.48

when comparing the material's tensile behaviour in gas and in liquid metal environment. Therefore, Fig. 2 shows the uniform and total elongation as function of temperature. In Fig. 2, the full black points represent the tensile results of SSRT tests at various temperatures, performed in liquid LBE environment while continuously bubbling with an argon and hydrogen gas mixture. The gray hollow symbols represent the SSRT results of the same bainitic elevated Si steel, tested at various temperatures using the same testing facility but in gas environment (Ar + 5% H_2). As mentioned before, all tests were performed at a strain rate of $5 \times 10^{-6}\ s^{-1}$.

The tensile behaviour of the bainitic Si enriched S2439 steel is significantly different in liquid LBE environment than in gas as can be seen in Fig. 2. At 150 and 200 °C, there is no change in the uniform elongation. The total elongation is however reduced by about 5%. This type of behaviour, where the uniform elongation remains the same while the total elongation is reduced was previously encountered in the liquid metal embrittlement investigation of the commercial ferritic-martensitic steel T91 in liquid lead–bismuth eutectic and the reduced activation steel Eurofer97 in liquid lead–lithium eutectic [22].

However, in contrast to the liquid metal embrittlement of T91 and Eurofer97 in LBE or LLE, where the maximum effect of the liquid metal environment on the tensile properties was a decrease in total elongation [22], there is a strong decrease in both uniform and total elongation of the bainitic Si enriched S2439 steel at 300 and 350 °C. As shown in Fig. 2, there is nearly no ductility left when the bainitic Si enriched S2439 steel is tested in liquid LBE environment at 300 and 350 °C. The total elongation of the tests performed in LBE at these temperatures is smaller than the uniform elongation at the same temperature when tested in gas environment. This indicates that the material does not reach its expected ultimate tensile strength when tested in liquid LBE between 300 and 350 °C.

Above 350 °C, the bainitic elevated Si steel S2439 recovers its ductility and at about 400 °C, the effect of the liquid metal environment seems to entirely disappear.

The mechanical behaviour observed in Fig. 2 is typical for solid metal/liquid metal couples which suffer from severe liquid metal embrittlement. As mentioned above, the temperature interval where the liquid metal environment affects the material's ductility is called the ductility trough. Based on Fig. 2, one can state that the ductility trough of the bainitic elevated Si steel S2439 seems to be between 250 and 375 °C. This means that in contrast to most observed cases of LME [11], the most severe embrittlement of steel S2439 is not near the melting point of the liquid (T_m = 123 °C). Compared to the LME observed in ferritic–martensitic steels such as T91 and Eurofer97, the effect of the liquid metal environment is more severe for the bainitic elevated Si steel S2439 and the onset of the ductility trough is at a lower temperature. The width of the ductility trough is slightly more narrow for the couple S2439 steel–LBE although it needs to be noted that the applied strain rate was lower than the one applied by Long et al. [17].

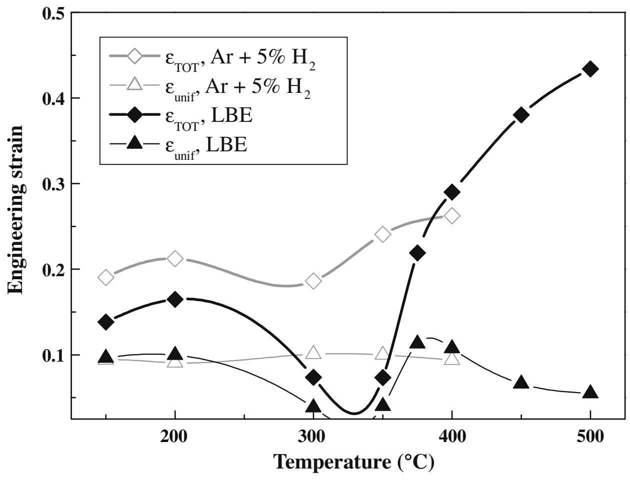

Fig. 2. Total and uniform elongation of bainitic elevated Si steel S2439 as function of temperature (strain rate: $5 \times 10^{-6}\ s^{-1}$). Full, black symbols for tests performed in liquid LBE while bubbling with Ar + 5% H_2; empty, gray symbols for tests performed in controlled gas atmosphere (Ar + 5% H_2).

When observing the fracture surfaces of the specimens tested in liquid LBE (see Fig. 3) it is clear that the LBE causes brittle, cleavage type fracture at 300 and 350 °C. In the upper two sets of fracture surfaces in Fig. 3, there is very little, if any, necking of the specimen prior to fracture. The close up of the fracture surfaces of the tests in LBE at 300 and 350 °C reveal clear river patterns in the grains of the bainitic structure. This is clearly an indication of transgranular cleavage fracture. It should be noted however that especially for the 350 °C specimen depicted in Fig. 3, there is still a certain fraction of dimpled fracture surface left. As can be seen in the close up picture, there are dimples in the areas near the grain boundaries. The occurrence of dimples in grain boundary areas while the insides of grains are clearly fractured in cleavage mode underlines the importance of grain orientation for LME to occur.

At more elevated temperatures, the sample shows an increasing amount of necking prior to fracture and furthermore the amount of dimples in the fracture surface increases. The sample tested in liquid LBE at 400 °C is fully dimpled which indicates full ductile behaviour. These fracture surface observations are in excellent agreement with the tensile behaviour of the bainitic S2439 steel previously discussed in Fig. 2.

To examine the embrittling effect of the liquid LBE at 350 °C more closely, we have depicted the stress–strain curves of the bainitic Si enriched steel in both LBE and gas environment together in Fig. 4. Despite a small deviation due to experimental scatter, there is no difference in elastic or plastic behaviour prior to fracture between the two tests. This confirms the findings of Westwood et al. [23] who stated that when LME occurs, the stress–strain behaviour of the solid is the same as in the unwetted condition until premature brittle failure occurs. Here, the bainitic S2439 steel suddenly broke after only about 7% of elongation when tested in liquid LBE while this steel is expected to have a total elongation of well over 20% at 350 °C. Furthermore, brittle fracture took place before the material could reach its ultimate tensile strength.

In Fig. 5, a comparison is made between the fracture surfaces of the test in controlled gas (Fig. 5A and B) and the test in liquid LBE at 350 °C (Fig. 5C and D). The fracture surface images in Fig. 5B and D are identical to those shown in Fig. 3 for the test at 350 °C. They are repeated here to stress the difference in fracture appearance between the test performed in gas and the test performed in liquid LBE environment. Fig. 5A clearly shows that the bainitic elevated Si steel S2439 shows a significant amount of plastic deformation prior to fracture when tested in gas. This ductile behaviour is confirmed by the close up depicted in Fig. 5B which shows a fully dimpled fracture surface.

When the same material is tested in liquid LBE under the same conditions of temperature and strain rate however, the material has very little ductility left as can be seen in the absence of necking in Fig. 5A. Furthermore, the river patterns in the close up image of the fracture surface (see Fig. 5B) strongly indicate transgranular brittle cleavage fracture.

No penetration of LBE into the matrix or along the grain boundaries was observed in any of the corrosion studies of elevated Si steels in contact with liquid LBE [6–10] and none of the observed embrittled fracture surfaces revealed intergranular fracture. This is in very sharp contrast with some reported cases of LME such

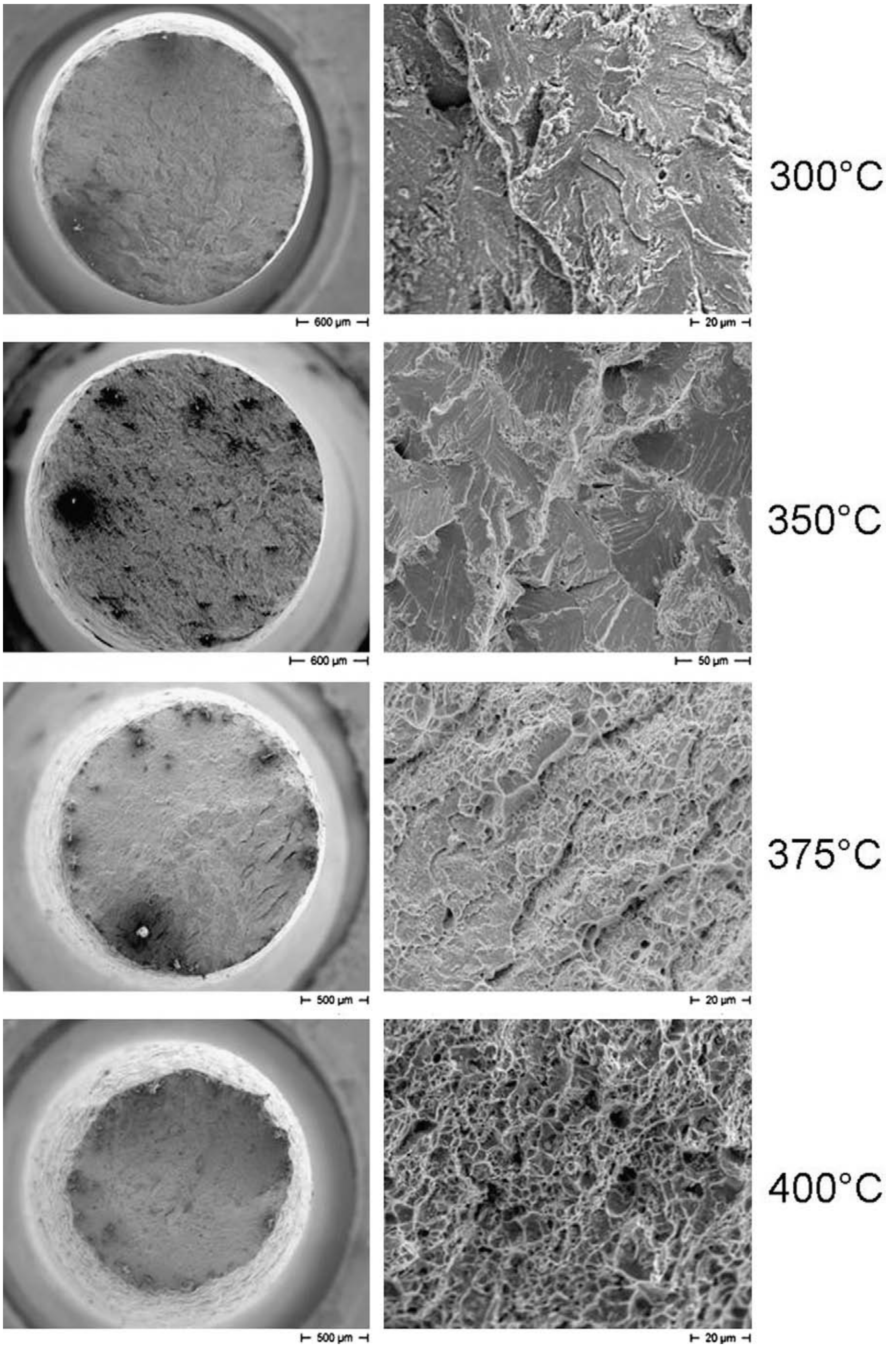

Fig. 3. SEM examination of fracture surfaces of S2439 steel tested in liquid LBE environment as function of temperature.

as for instance Al–Hg [23] and Al–Ga [23] and Ni–Pb [24], Ni–Bi [25] where the liquid metal is reported to penetrate the solid along the grain boundaries.

Based on the observations made of the LME of the bainitic elevated Si steel S2439, the adsorption induced reduction in cohesion model by Stoloff [27], Johnston, Westwood and Kamdar [23,26] seems to be the most likely model to explain the phenomenon of LME in contact with LBE observed in this study. This model states that the presence of liquid metal atoms at the crack tip lowers the energy barrier that needs to be overcome to separate the two atoms at the crack tip. Macroscopically, this model therefore claims that the presence of liquid metal at the crack tip lowers the critical cleavage stress.

By decreasing the cleavage stress, the presence of liquid metal would increase the DBTT temperature of the material. This shift would then define the upper temperature of the ductility trough. Since the actual cleavage stress of the S2439 steel in LBE or in gas environment could not be measurement in this work, it would not be possible at this point to firmly confirm the proposed mechanism.

The lower temperature of the ductility trough is thought to be defined by the ability of the liquid metal atoms to reach the crack tip and their influence on the cohesive energy as a function of temperature. This might explain why for the majority of embrittling couples, the LME is very dominant near the melting temperature. However, in the case of the S2439 steel in contact with LBE studied here and the ferritic–martensitic steel T91 in contact with LBE studied by Long et al. [17], the lower limit of the ductility trough is well above the melting point of the LBE. This may indicate a more complex set of parameters defining the lower boundary of the ductility trough than commonly assumed.

Electronic structure calculations on the surface energy reduction of pure iron by adsorption of LBE have shown there will be a reduction in the surface energy by between 16% and 35% depending on the crystallographic orientation (22% for (0 0 1)-plane; 35% for (0 1 1)-plane; 16% for (1 1 1)-plane) due to adsorption of LBE [28]. Although these values confirm a decrease in the cleavage stress due to a reduction in surface energy of the cleavage plane (the (0 0 1)-plane) it is unclear whether this is sufficient to explain the observed LME. The adsorption induced reduction in cohesion has not yet been confirmed by any structure calculations showing a decrease in cohesion of the atoms which can account for an additional decrease in cleavage stress. Furthermore, the available electronic structure calculations are based on pure iron in contact with LBE. Although many investigated steels are mainly comprised of iron, their mechanical behaviour in contact with LBE is clearly different [29].

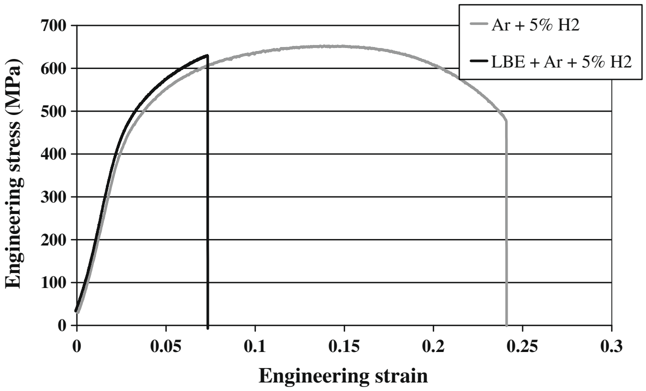

Fig. 4. Tensile curves of S2439 steel tested at 350 °C, using a strain rate of $5 \times 10^{-6}\ s^{-1}$ in Ar + 5% H_2 (gray) and in liquid LBE purged with Ar + 5% H_2 (black).

4. Conclusions

The tensile behaviour of an experimental bainitic silicon enriched steel in liquid lead–bismuth eutectic environment was examined as function of temperature at a constant strain rate of $5 \times 10^{-6}\ s^{-1}$. To assess the effect of the liquid metal environment, comparison was made with similar tests on the same material performed in an argon and hydrogen gas mixture.

Based on the observations discussed in this paper we can state that despite the improved corrosion resistance of elevated Si steels in contact with liquid LBE, the bainitic S2439 steel was very prone to liquid metal embrittlement between 300 and 375 °C with the transition in mechanical behaviour from ductile to predominantly brittle between 200 and 300 °C. At 300 and 350 °C, the material broke by brittle cleavage fracture with nearly any prior plastic deformation. Above about 400 °C, the material fully restored its normal ductile behaviour.

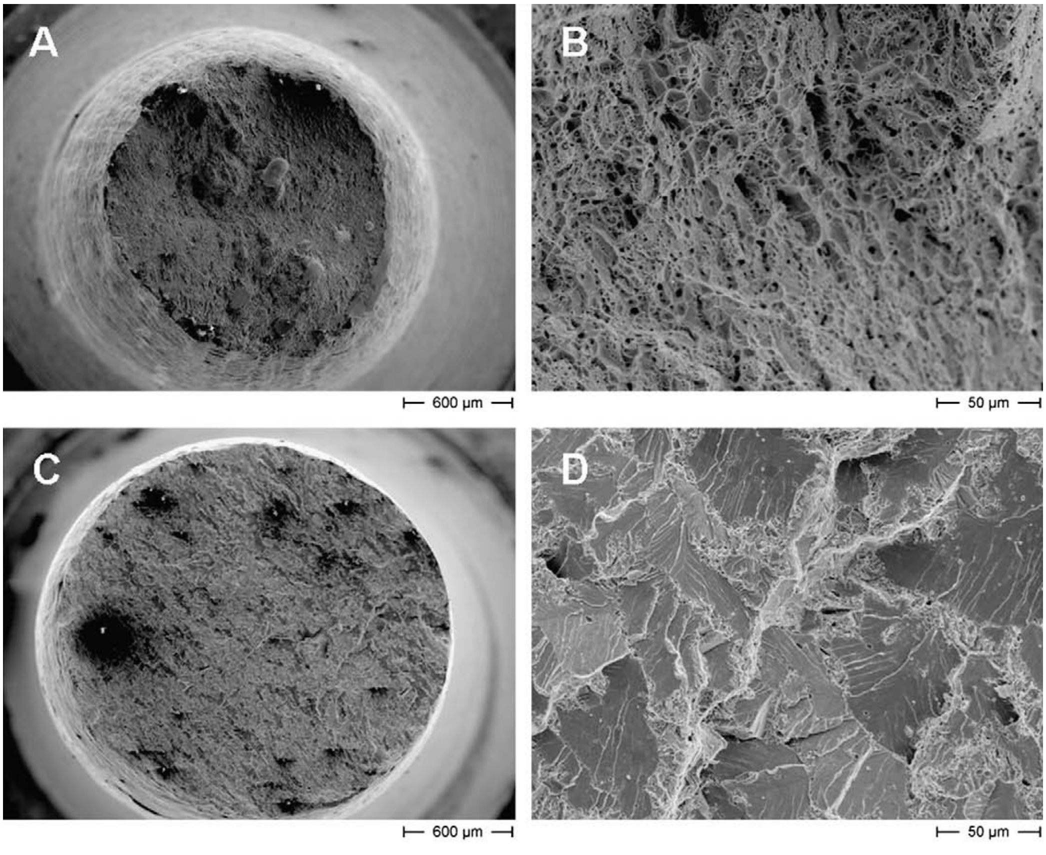

Fig. 5. SEM examination of fracture surfaces of S2439 steel, tested in Ar + 5%H_2 and LBE at 350 °C, using a strain rate of $5 \times 10^{-6}\ s^{-1}$. (A) Overview of S2439 tested in Ar + 5% H_2 showing significant necking. (B) Detail of fracture surface depicted in A, showing a fully dimpled surface indicating ductile fracture. (C) Overview of S2439 steel tested in LBE purged with Ar + 5% H_2 showing no necking; D: Detail of fracture surface depicted in C, revealing brittle fractured grains showing river patterns and a small fraction of dimples on certain grain boundary areas.

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