

Effect of preliminary diffusion oxidation on mechanical properties of ferritic steel in oxygen-containing lead



O.I. Yaskiv*, I.S. Kukhar, V.M. Fedirko

Physical-Mechanical Institute of National Academy of Sciences of Ukraine, 5, Naukova St., Lviv 79601, Ukraine

HIGHLIGHTS

- We investigated the influence of pre-oxidation on mechanical properties of ferritic steel (Fe-11Cr) in oxygen containing lead melt (10^{-6} – 5×10^{-8} wt.%) at temperatures range 400–600 °C.
- We revealed that an increase in oxidation temperature leads to the increase in grain size and evolution of oxide's composition from magnetite to chromium-containing spinel and discontinuous formation of oxide Cr_2O_3 .
- The presence of oxide layer limits direct contact between metal and lead that consequently prevents steel's embrittlement.
- The pre-oxidation which minimally affects on the grain sizes of steel (600 °C and 800 °C within 24 h) provides the minimal losses in plasticity in lead.

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ABSTRACT

The influence of preliminary diffusion oxidation on mechanical properties of ferritic steel SUH409L (Fe-11Cr) in oxygen containing stagnant lead melt (10^{-6} – 5×10^{-8} wt.%) in the temperature range 400–600 °C has been studied. Oxidation in the temperature range 600–800 °C for 24–150 h results the formation of thin oxide films (1–4 μm). The thickness and composition of films depend on temperature and duration of oxidation. In particular, the increase in temperature leads to the evolution of oxide's phase composition from magnetite to chromium-containing spinel and discontinuous formation of chromium oxide Cr_2O_3 . Another effect of heat treatment during oxidation is the growth of grain size of steel with increasing temperature and duration. The oxidation leads to the decrease of tensile strength and ductility of steel that correlates with increasing grain size. In lead the strength of pre-oxidized steel reduces. It manifests more significant for oxidation modes which lead to maximum growth of grain sizes. It was proved that the presence of oxide layer limits direct contact between metal and lead that consequently prevents steel's embrittlement. The oxidation which minimally affects on the grain sizes of steel (600 °C and 800 °C for 24 h) provides the minimal losses in plasticity in lead.

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1. Introduction

Lead due to the high nuclear and thermo-physical properties is considered as neutron multiplier in the fusion blanket and as candidate coolant and target materials for lead alloy cooled fast reactors [1–3]. Ferritic-martensitic steels are characterized by high yield strength, low coefficient of thermal expansion and good thermal conductivity and therefore find wide application as reactor materials for the first walls, blankets and fuel elements of fast reactors.

Corrosive damage and liquid metal embrittlement remain the most important issues in the system “solid–liquid metal”. The corrosion mechanism depend on oxygen concentration and in

case of the low concentration is different from the oxidation type corrosion [4]. To minimize corrosion degradation and promote the formation of protective oxide layer Me_3O_4 (Me = Fe, Cr) which prevents direct contact between metal and melt the concept of active oxygen control in melt which is maintaining low levels of oxygen in the liquid metal ($\sim 10^{-6}$ – 10^{-7} wt.% O) currently considered as the most optimal [5–7]. However, such coatings have uneven growth of interface, are heterogeneous and may contain cracks between sub-oxide layers. Therefore they can flake off with time as a result of mechanical destruction in flowing melt (erosion) or by lowering the protective properties due to the evolution of phase-structural state during long exposures [8].

Another way to protect the steel's surface in lead can be targeted modification of phase-structural composition of the surface layer by preliminary oxidation. Pre-oxidation allows to avoid the dangerous initial stage of interactions between unprotected

* Corresponding author.

E-mail address: oleh.yaskiv@ipm.lviv.ua (O.I. Yaskiv).

Table 1
Conditions of pre-oxidation and mechanical tests.

Pre-oxidation						
Temperature (°C)	600	700	800			
Time (h)	24	150	24	150	24	150
Environment	Argon dotted with 20 wt.% O					
Mechanical tests						
Temperature (°C)	400–600					
$C_{O Pb}$ (wt.% O)	10^{-6} – 5×10^{-8}					
Tensile rate (mm/s)	0.08					
Strain rate (s^{-1})	5×10^{-4}					

steel surface and lead, where the solution dominates and then double-layered magnetite based oxide is being formed intensively.

In general, the mechanisms of formation, microstructure characteristics and protective properties of scales depend on the composition of saturating medium, temperature of oxidation, surface characteristics and chemical composition of the steel [9].

The positive effect of pre-oxidation on corrosion resistance of steel in liquid metals is stated in [10–14]. For instance, in [10] the effect of pre-oxidation in water vapor and air (773 K; 12, 24 and 72 h) on the corrosion behavior of steel in fluid of oxygen-containing eutectic Pb–Bi ($V=1$ m/s, 823 K, $C_{O|Pb-Bi} = 1.7 \times 10^{-8}$ wt.%, 500 h) was studied and it was showed that pre-oxidation is effective for reducing corrosion losses and prevention of the dissolution of steel in the initial stages of interaction as compared to unoxidized steel. In [13] the corrosion resistance of martensitic steel F82Hmod after pre-oxidation in air at 600 °C for 2 h in Pb–Bi at 400 and 600 °C under conditions of dissolution (“pure” melt) and “oxidation” (10.8 wt.% O) was showed that at low oxygen concentrations in melt the pre-oxidation retards, but does not prevent corrosion of steel. Moreover, when oxygen concentration in melt is sufficient to oxidation prevails under dissolution, the pre-oxidation promotes the growth of homogeneous and corrosion resistant oxide layer. In [14] reported that after gas oxidation of EP823 steel the chromium oxide was formed and it was showed that this oxide is an effective barrier to the diffusion of iron ions, slowing down the growth of bi-layered magnetite scale in melt even with high oxygen potential.

At the same time in [15] it was reported that thin pre-formed oxide film ($\leq 1 \mu\text{m}$; AISI 316 steel was pre-oxidized in air at 800 °C

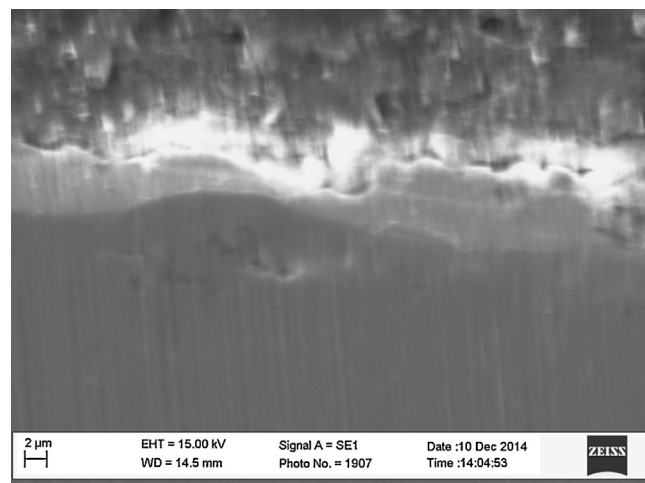


Fig. 1. Cross-section of Fe-11Cr steel after pre-oxidation at 800 °C for 24 h.

for 60 h) did not play a role of efficient protective barrier to further oxidation in the oxygen enriched Pb–Bi melt at 550 °C for 1335 h. In [12] reported that regardless the thickness of pre-formed oxide the subsequent growth of scale (magnetite) in melt was revealed.

Thus, it can be assumed that steel protection against corrosion in lead melts by means of pre-oxidation in air or other oxygen-containing environments increases the time of beginning the destructive processes of corrosion.

It is expected that the pre-formed oxide layers may not only protect against corrosion but also prevent liquid metal embrittlement (LME) of steels. For instance, in [16,17] it was shown, that the embrittlement of T91 steel in Pb–Bi depends on the conditions of the contacting surfaces: i.e. in the case of indirect contact (through the oxide film) a ductile fracture takes place; if barriers are removed and direct contact is provided, then the transition from ductile to brittle fracture occurs. At the same time, the authors [18,19] have studied the effects of oxidation on LME manifestations in Pb–Bi and stated that brittle fracture, which provoked by liquid metal, may occur even with the presence of pre-formed oxide film. Therefore, any type of oxide films, even if it makes impossible to direct contact

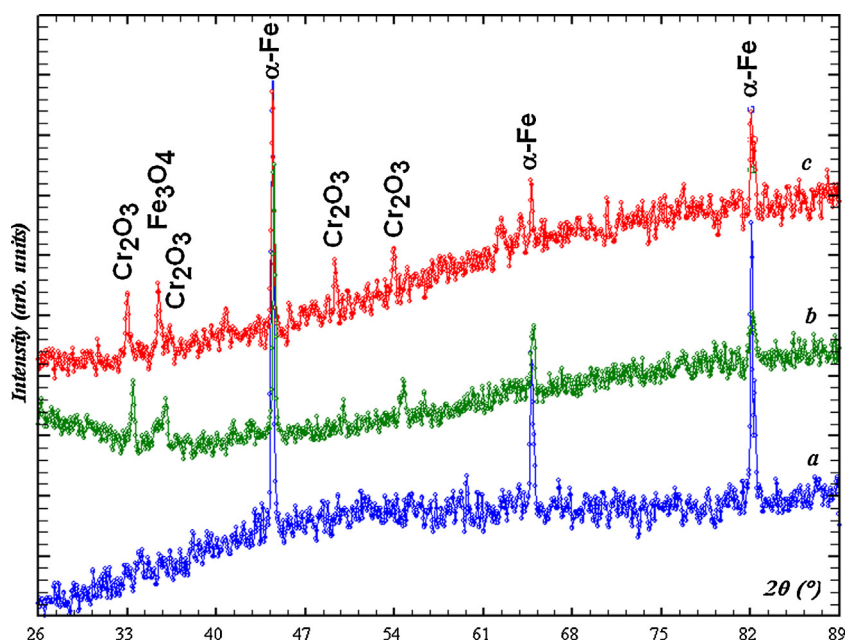


Fig. 2. XRD patterns from Fe-11Cr steel after pre-oxidation in argon-oxygen gas mixture with 20 wt.% O: 600 °C, 24 h (a), 600 °C, 150 h (b) and 800 °C, 24 h (c).

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