

### L.3: Improvement of oxidation resistance of P91 grade steel by laser shock peening

9Cr-1Mo based steel (P91) has been chosen as a candidate material for structural components in Gen-IV nuclear reactors and ultra-supercritical power plants due to its excellent mechanical properties, high thermal conductivity and low thermal expansion. However, its use is limited at temperatures above 550 °C due to poor oxidation resistance. Improvement of oxidation resistance by increasing Cr content beyond 12 wt.% is not feasible because it leads to deterioration of mechanical properties. In the present study, an attempt has been made to improve the oxidation resistance of P91 steel by creating nano grains near surface using laser shock peening (LSP).

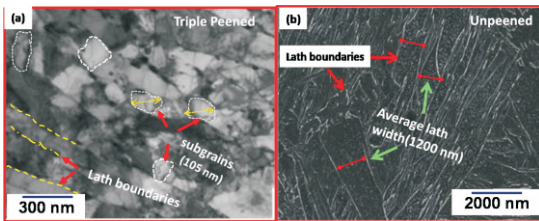


Fig. L.3.1: (a) TEM micrograph taken at near surface (20 μm from top) of triple peened sample, (b) SEM micrograph of unpeened P91 sample.

P91 samples of cuboid shape were shock peened on all the sides using an in-house developed Q-switched Nd:YAG laser at laser power density of ~3.5 GW/cm<sup>2</sup> for single and triple LSP impacts, while fixing other peening parameters such as laser spot size (3 mm), repetition rate (2 Hz) and overlap percentage (70%). Effect of LSP on near surface microstructural modification in P91 grade ferritic-martensitic steel has been studied using transmission electron microscopy (TEM) technique. The lath structure at near surface (~20 μm depth from top) underwent severe refinement in the samples subjected to multiple LSP impacts. From Figure L.3.1(a), it is clear that in the triple peened samples, well defined polygonal sub-grains of size about 105±10 nm have been formed with respect to unpeened sample, which had lath width of the order of ~1200 nm (refer Figure L.3.1(b)). In the case of single peened samples, lath width was found to be of the order of ~300 nm. Further, to study the effect of nano-grain microstructure on oxidation behaviour of the P91 steel, unpeened, single peened, and triple peened samples were exposed to air atmosphere at 700 °C in a muffle furnace for the time duration varying from 5 to 200 hours. It is found that oxidation rate in single and triple peened samples has been significantly reduced compared to unpeened samples. This can be witnessed in Figure L.3.2, where the photograph of oxidized surfaces of unpeened, single peened and triple peened samples for 20 and 40 hrs. are presented. It is clear that in the case of triple peened samples,

oxidized scale is very feeble in comparison to single and unpeened samples. Further, to quantify the actual oxidation rate in all the three samples, variation of specific weight gain as a function of time has been measured using a high precision micro-balance and corresponding result is shown in Figure L.3.3.

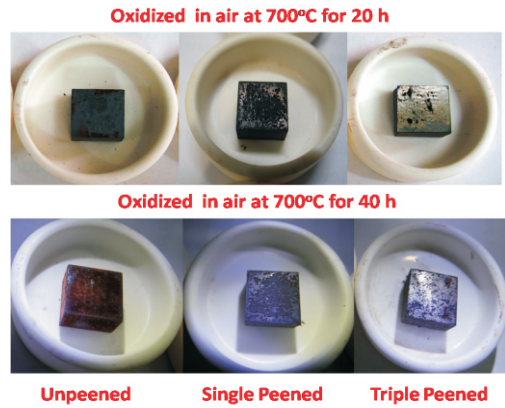


Fig. L.3.2: Photograph of unpeened, single peened, and triple peened P91 samples oxidized at 700 °C in air for 20 and 40 hrs.

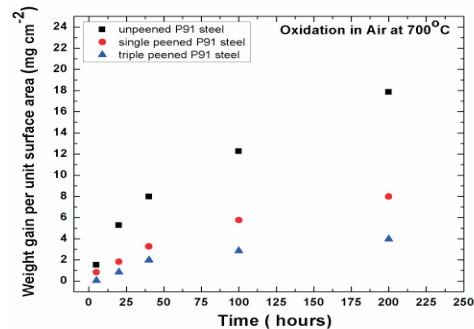


Fig. L.3.3: Variation of specific weight gain as a function of time of unpeened, single peened, and triple peened P91 samples in air at 700 °C.

It is observed that even after 100 hours of heating, the specific weight gains were about 15.27, 6.75, and 3.08 mg cm<sup>-2</sup> for unpeened, single peened, and triple peened samples, respectively. The results confirm that with respect to unpeened P91 sample, oxidation rate in single and triple peened samples were reduced by two and five fold, respectively. The enhancement in oxidation resistance of LSP treated samples is attributed to the increased diffusivity of Cr due to the availability of significantly enhanced grain boundary area associated with nanograins. Faster Cr diffusion to the laser peened surface facilitated formation of protective layer of dense Cr<sub>2</sub>O<sub>3</sub>, which provided protection against further oxidation. This work has been carried out in collaboration with LMPD, RRCAT.

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