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Thermal Effects on Thin Laser-Peened Ferritic-Martensitic Samples

M. Caro, T. Zalesky, P. Hosemann, B. S. El-dasher, W. G. Halsey, B. Stuart

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Abstract

Laser peening is emerging as a promising technique to improve the corrosion resistance of cladding material candidates for lead-cooled fast reactors (LFRs). The challenge is in the performance capability of ~ 1 mm-thick fuel-pin cladding. Ferritic-martensitic (F/M) steels are foreseen as possible candidates that stand severe conditions of high dose (150 dpa), and high temperature (~500-600°C) under the corrosive environments of molten lead or lead-bismuth. In this paper, we present the results of experiments carried on laser peened (LP) samples of F/M steels HT9, T91, EP823, as well as the austenitic material 316 L. The samples underwent a thermal treatment in an oven at 520°C, and XRD compressive stress results indicate that the F/M samples do not retain the residual stress after 2 weeks of heat treatment. The corrosion behavior in flowing lead-bismuth eutectic (LBE) at 535°C has been investigated as well. Also, irradiation experiments of LP samples to a maximum dose of ~10 dpa are foreseen.

Introduction

The structural and cladding material for lead-cooled fast reactor (LFR) systems must be corrosion resistant in lead or lead-bismuth eutectic (LBE) coolants. Laser-peening technology is emerging as a promising technique to enhance corrosion-resistance of materials that must withstand high operating temperatures and high fast flux during a large number of years. The laser peening (LP) treatment generates a compressive stress state altering the metal oxide bonds on the surface. This advanced surface treatment can potentially retard the corrosion of LFR structural materials.

At LLNL, LP was applied to several very thin (~ 0.8 mm thick) samples of F/M steel. The materials tested correspond to selected steels (HT9, T91, EP-823, and 316L), the nominal compositions of which are listed in Table 1. Each coupon (0.8mm x 8mm x 35mm) was treated on both sides and only the upper halves of all the coupons were laser peened, see Fig. 1.

Material	C	Si	Mn	S	P	W	Cr	Ni	Mo	V	Nb	N	Ti
EP823	0.18	1.05	0.6	---	0.012	0.65	11.4	0.7	0.67	0.4	0.2	---	0.03
316L	0.035	0.08	2	0.03	0.04	---	16	10	2	---	---	---	---
HT-9	0.2	0.25	0.5	----	----	0.5	12	0.56	1	0.3	---	---	---
T91	0.1	0.4	0.45	----	-----	-----	9	---	1	0.2	0.08	0.05	---

Table 1: Nominal chemical composition of the coupons investigated.

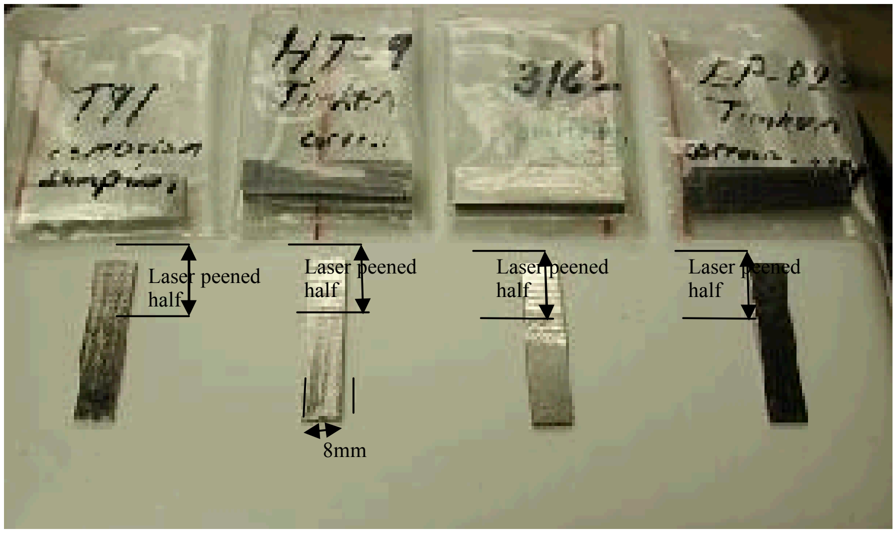


Fig. 1: T91, HT9, 316 L and EP-823 samples after laser peening.

Some of these samples were sent to LANL to participate in corrosion tests carried out at the LBE DELTA Loop. In the Delta Loop the samples were exposed to flowing (~1 m/s) LBE at 535 ^{o}C for ~200 h, ~ 400 h, and ~600 h. SEM imaging and studies to determine the composition and oxidation state of the species in the scale were performed at LANL. The goal of these studies is to evaluate the improvement of the corrosion behavior due to laser peening and the corrosion/oxidation mechanism which take place in this environment. The effect of high temperature on the induced LP residual stress was a concern for these corrosion experiments. In the present paper we report results of experiments conducted at LLNL giving insight on thermal treatment effects on very thin LP T91 samples.

Background Information

Recent experiments at LLNL have shown that laser peening can virtually eliminate occurrence of stress corrosion cracking in metal [1, 2]. Prior to this experiment, there has been very limited investigation into LP parameters for thin materials (<3mm). The data from a former study in a titanium alloy was used to approximate the depths of compressive stress which could be achieved in the cladding materials. This previous experiment used titanium alloy coupons (Ti, 6%Al, 4%V) with a 3 mm thickness to determine the magnitude and depth of residual stresses induced by laser peening [3]. The ultimate tensile strength for this material is ~900 MPa and the tensile yield strength is ~800 MPa, with variations dependant on the heat treatment. Residual stress was measured using the slitting method and reported as a function of depth through the samples as shown in Fig. 2.

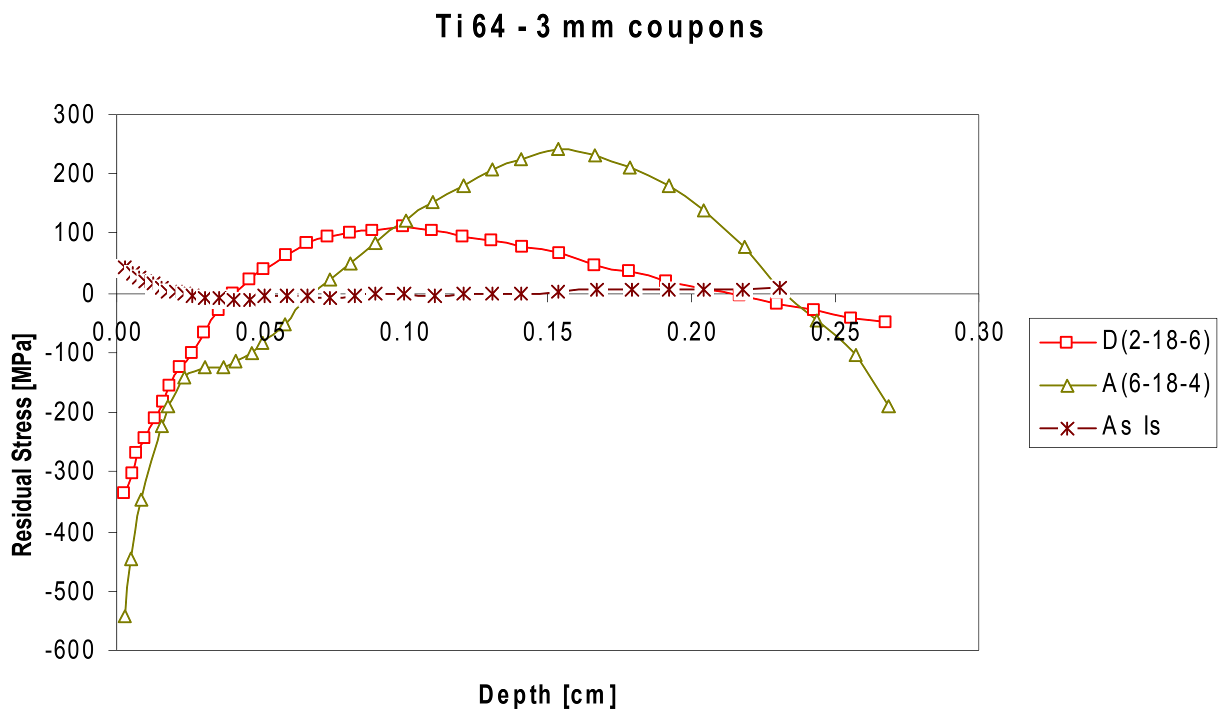


Fig. 2: Residual compressive stress profile for a 3mm thick titanium alloy coupon [3].

The "As Is" line (star) corresponds to the sample without laser peening, A(6-18-4), i.e. (6 GW/cm^2-irradiance, 18 ns – laser pulse duration, 4 layers), and D(2-18-6) show that the RS depth has a dependence on the number of peening layers and the level of irradiance. Note that a "layer" here refers a 100% surface coverage with laser spots. Subsequent treatment layers are offset from 33% to 50% in both axes to provide uniform coverage.

Results of this measurement demonstrate that lower laser irradiances induce lower magnitudes of residual stresses. In the 3mm thick titanium coupons, the depth of compressive residual stress reaches approximately 20% of the coupon thickness. For typical cladding thicknesses of 1mm, our objective would be to induce the residual compressive stress to reach a dept of approximately 0.2mm (200 µm for the corrosion samples to conform to ASTM standards).

Corrosion Coupon Treatment

The corrosion specimens were treated with 2 layers of LP with each layer providing 100% surface coverage and a 50% overlap in both horizontal and vertical directions with respect to the previous treatment layer.

Once the laser parameters were calibrated 4 samples of 316L, 4 HT9, 4 T91, and 4 EP-823, were peened on both sides and sent to the Delta Loop for the corrosion tests. The samples were peened to half height level (see Fig.1) using laser treatment parameters obtained in the Preliminary Test. Laser peening parameters for sixteen coupons (316L, HT9, T91, and EP823) are 8GW/cm^2 irradiance, 18 ns pulse width, and 2 layers of treatment (8-18-2).

Thermal Treatment Effects on Almen Coupons

Some very limited work on LP coupons suggests that the stress imparted by LP does not show significant relaxation with time and temperature [4]. This was shown for testing temperatures of 150 ºC and time intervals of 3 weeks. To test the case of higher temperatures (~520 ºC) and longer exposure times (~600 h) five samples of "Almen C" strip samples were LP under the same laser parameters obtained in the "Preliminary Test" and then placed in an oven at 520 °C. (Note that this time the samples were laser-peened on one side only). Two samples were removed after one week and the other two samples were removed after two weeks of heating.

Qualitative residual stress (RS) measurements were done by determining the deflection of the sample. The deflection was measured at several instances (~ 1 d, ~ 2 d, ~ 6 d, and ~ 7 d) and reported as a function of time (see Fig. 3 a: results for the 5 coupons tested and b: average value).

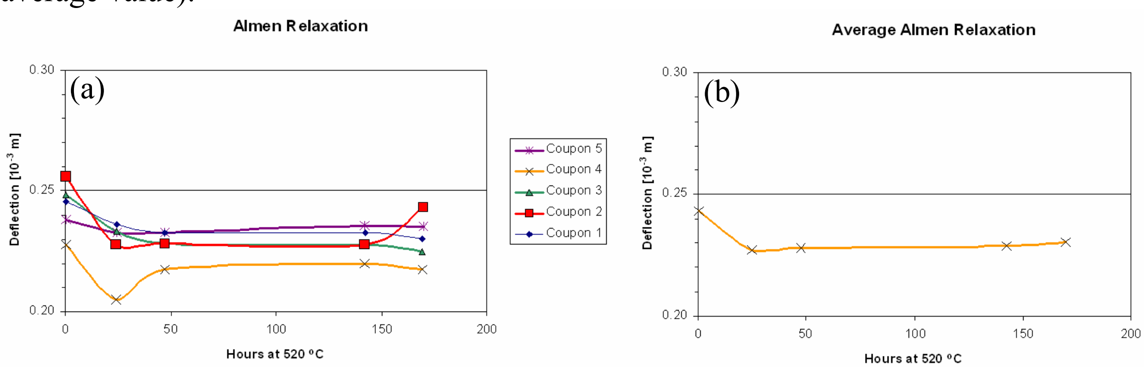


Fig. 3: Thermal effects on laser-peened "Almen_C" strip samples.

The experiment showed that the stress induced on the 1mm-thick laser-peened samples remained approximately constant after a heat treatment of one week. The thermal treatment seems to affect the laser-peened sample in the first 2 days, after which the induced stress remains constant. This process will also take place at the DELTA Loop where in addition to the thermal affects, the corrosion process will start immediately. Further studies are needed to understand the kinetics of both processes and their interaction. Also it is important to characterize the sample initial conditions (influence of pre-oxidization if done) because these parameters might affect the final sample response.

Residual Stress Measurements

Laser peening adds a significant amount of residual stress to the surface of the sample. The amount of RS added is difficult to predict. It depends on the laser peening parameters and the specimen geometry. Therefore, two residual stress measurement techniques were used in this project: x-ray diffraction (XRD) and Slitting Method. The slitting method uses metallic foil strain gauges to measure the strain released during incremental cuts into the depth of the material [9]. This was performed by Mike Hill's Material Performance group at UC Davis. Initially, these measurements were going to be complemented with x-ray diffraction to get surface stresses, as this would have contributed to solve the difficulty with slitting, i.e. the absence of data at the surface.

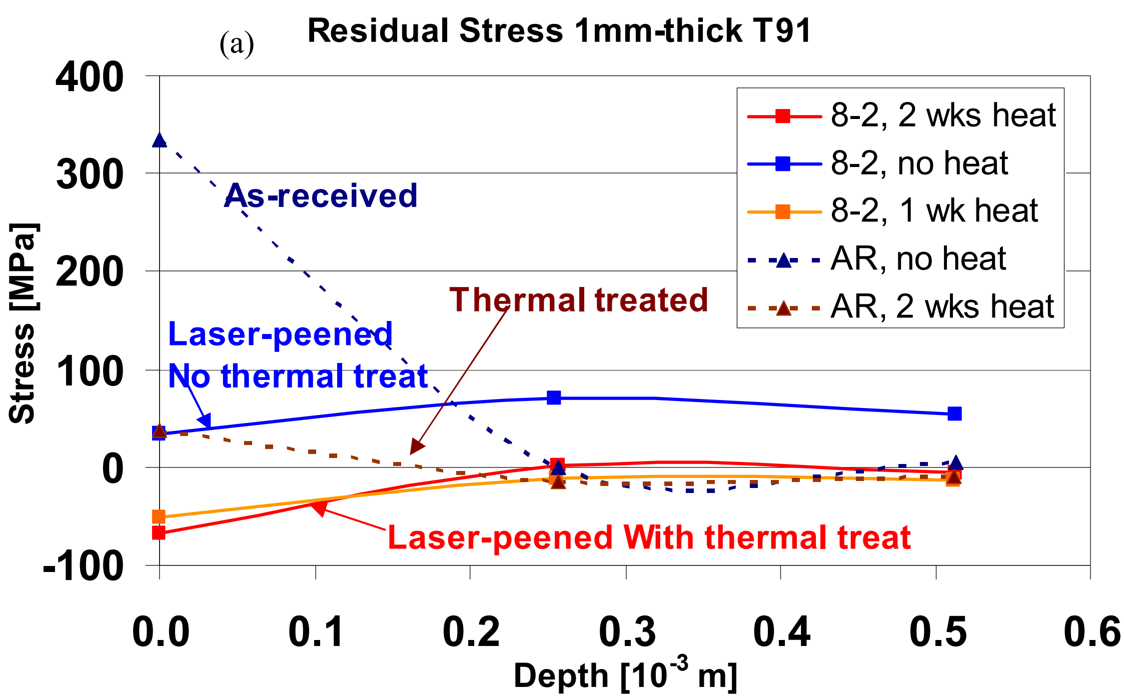
Residual Stress Measurements Using XRD Method

Residual stress (RS) is measured as a function of depth for this experiment was measured using x-ray diffraction (XRD). Thermal effects on 1-mm thick T91 laser-peened samples were investigated. Five samples were tested for thermal relaxation (see Table 2)

Type	Laser Peening (8-18-2)	Heat Treatment (520 °C)
CASE 0	None	None
CASE 1	LP	1 week
CASE 2	LP	2 weeks
CASE 3	LP	None
CASE 4	None	2 weeks

Table 2: T91 test matrix for XRD measurements

The samples were placed in an oven preheated to 520 °C and RS relaxation measurements using X-ray diffraction were done after one week and 2 weeks of heating. RS relaxation results corresponding to the five T91 coupons tested are presented in Fig. 4a, and Fig. 4b.



(b)

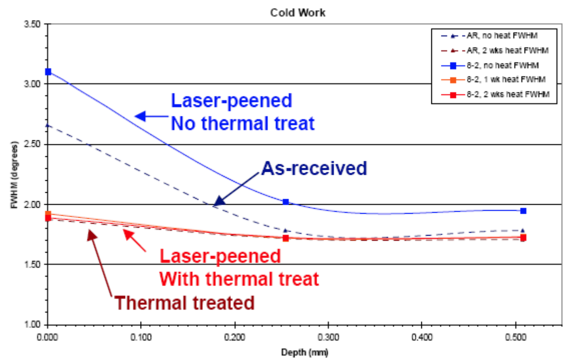


Fig. 4: a) Residual-stress versus depth b) Cold-work measured in terms of FWHM for laser-peened (LP) and un-peened as-received (AR) T91 steel samples without thermal treatment (no heat) and with 1 week and 2 weeks of thermal treatment (1 wk heat, 2 wks heat).

Two data points were obtained from each coupon at three different depths (surface, 0.25 mm, 0.5 mm). The data points are averaged and a line guide-to-eye is drawn to connect the points. Fig. 5 shows the results for the As-Received (AR) sample, i.e. the sample that was not laser peened and had no thermal treatment applied. The objective of laser peening the AR sample is to generate a residual compressive stress through about 20% of the sample thickness. The samples that participated in the corrosion test were laser peened on both sides and might show a larger compressive stress than the induced stress apparent in Fig. 5.

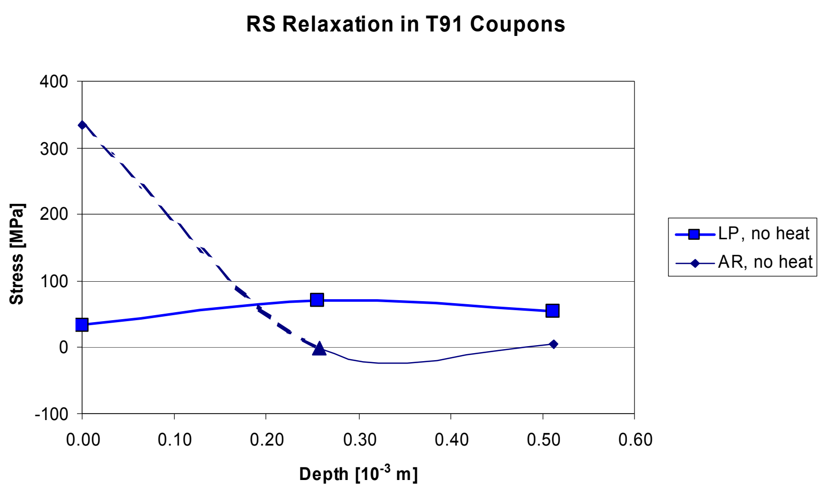


Fig. 5: Laser-peening effect on the 1-mm thick T91 coupon.

The tension in the sample relaxes after the thermal treatment is applied on the non laser peened coupon (AR, 2 wks heat at 520 ^{o}C), Fig. 6. Further studies should consider laser-peening the sample after the thermal treatment is applied as this was not done here. All the laser-peened samples were first laser peened and then underwent thermal treatment.

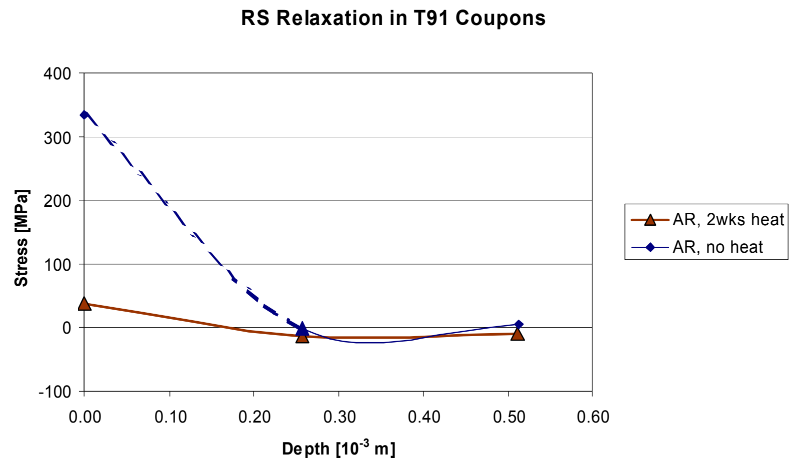


Fig. 6: Thermal effect on the 1-mm thick T91 un-peened coupon

The thermal treatment effect on the laser-peened sample is shown in Fig. 7. Taking into account that error bars associated with the data points are big (~ 100 MPa, not shown in the figure) we conclude that RS relaxation results are similar for heat treatments after 1 or 2 weeks.

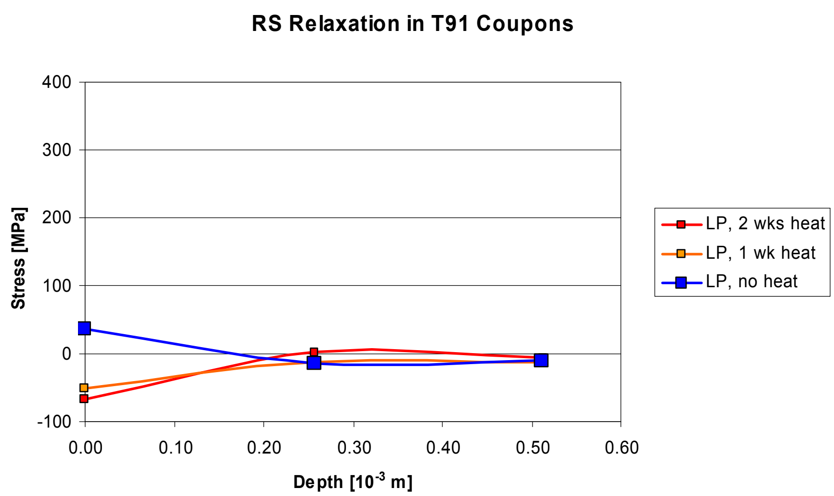


Fig. 7: Thermal effect on the 1-mm thick T91 laser-peened coupons

As shown before for the case of the Almen strips, the sample is not affected any more by thermal treatment after the first two weeks. This should also be the case of the samples in the DELTA loop corrosion test after 3.5 weeks (600 h) of operation.

Note that the samples have been laser peened on half of the surface to compare the effect of corrosion on the un-peened and peened regions. If a RS experiment is done on the samples that participated in the corrosion test we expect results similar to those plotted below in Fig. 8. Several samples that participated in the corrosion test have been shipped to LLNL and RS measurement will be performed.

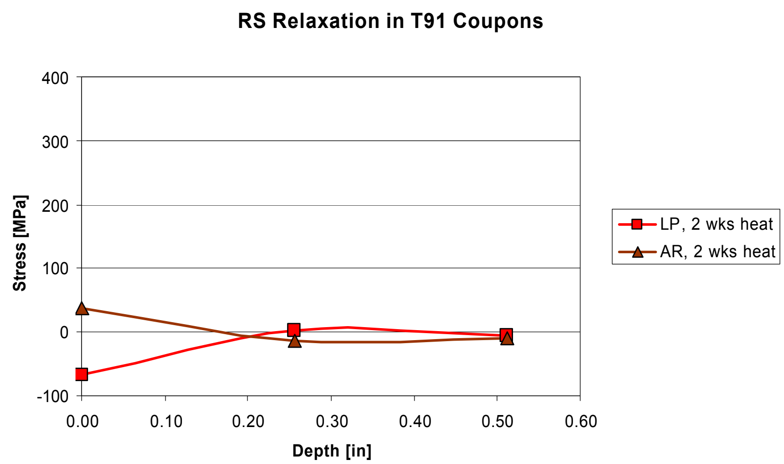


Fig. 8: Thermal effect on un-peened and peened T91 samples.

XRD measurements show large errors in small levels of residual stress (RS), and are difficult to interpret. Further measurements of residual stress were done using the slitting method, as addressed in the following section.

Residual Stress Measurements Using Slitting Method

The residual stress in the bulk of the coupon was measured using the slitting method. See Table 3 for the coupons measured using the slitting method.

SN	Mat'l	t (mm)	Laser-peening Treatment Irradiance - # layers	Heat (1wk)
2-LH-HT9	HT9	1	LP 8-2	500C
1-HH-HT9	HT9	1	LP 8-2	600C
3-NH-HT9	HT9	1	LP 8-2	none
4-NH-HT9	HT9	1	None	none
2-LH-316L	316L	1	LP 8-2	500C
1-HH-316L	316L	1	LP 8-2	600C
3-NH-316L	316L	1	LP 8-2	none
4-NH-316L	316L	1	None	none
5-NH-316L	316L	3	LP 6-2	none
4-NH-316L	316L	3	LP 8-2	none
3-NH-316L	316L	3	LP 10-2	none
1-LH-316L	316L	3	LP 10-2	500C
2-HH-316L	316L	3	LP 10-2	600C

Table 3 Experimental parameters for residual stress coupons

Due to the difficulty in obtaining sufficient material, EP823 and T91 coupons were not included in the slitting residual stress measurements. Thicker coupons of 3mm were measured in 316L to more accurately determine the reduction in residual stress due to the heating process.

To duplicate the laser peening treatment in the residual stress coupons, the same laser parameters were used. The residual stress specimens were treated with 2 layers of LP with each layer providing 100% surface coverage and a 50% overlap in both horizontal and vertical directions with respect to the previous treatment layer. Laser peening parameters for the 1mm coupons (316L and HT9) are 8GW/cm^2 irradiance, 18 ns pulse width, and 2 layers of treatment (8-18-2). Both sides of each coupon were treated. The 316L 3mm thick coupons were treated with a slightly higher irradiance (10GW/cm^2) to induce more residual stress.

The residual stress data in Fig. 9 seems to show two trends. First, the laser peened coupon with no heat treatment (blue) shows a tensile stress at the surface. The tensile peak at the coupon surface is different than expected as these coupons were all cut using a wire EDM and then polished to remove the recast layer. Regardless, the stress seems to relax in the laser peened samples during both heat treatments (orange and red). Second, reducing the laser treatment intensity seems to generate more residual stress. The highest level of residual compressive stress is obtained with the 6 GW/cm^2 treatment (pink), leading the authors to believe that the 1mm coupons may have been laser peened "too hard".

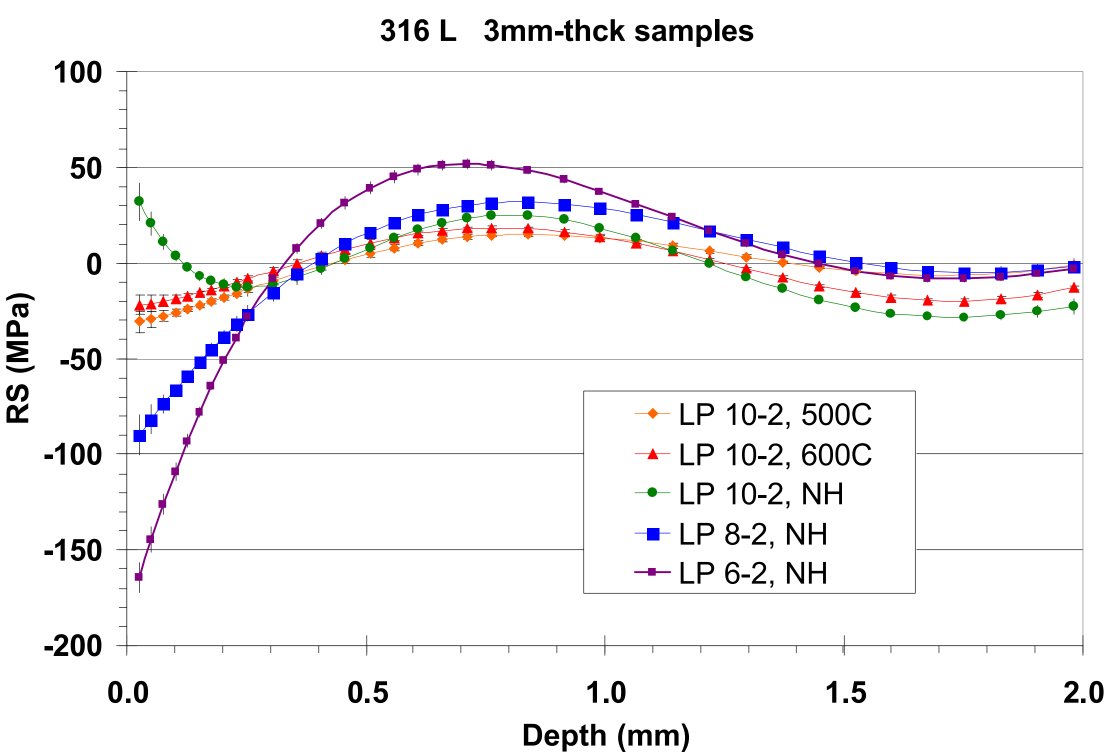


Fig. 9: Residual stress measurements for 3 mm thick 316L coupons.

It is unclear what conclusions can be drawn from the 1 mm data as the results shown in Fig. 10 and 11 below seem inconsistent between the two materials (316 L and HT9). Again there seems to be an unexpected amount of residual stress remaining from the machining process (purple curves). These coupons were wire EDM cut from plate material and then polished by hand to remove the EDM recast layer. This process had been expected to induce the least amount of residual stress to the coupons, however the data seems to suggest otherwise.

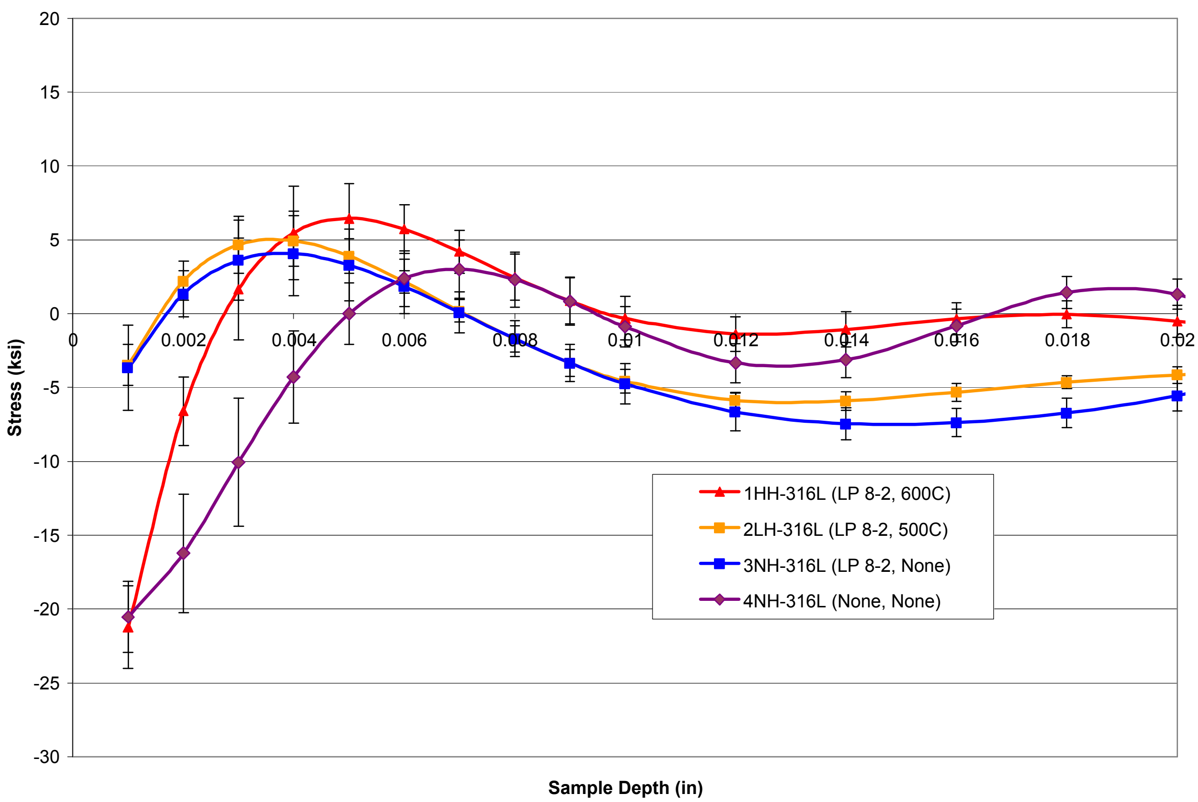


Fig. 10: Residual stress measurements for the 1mm thick 316L coupons.

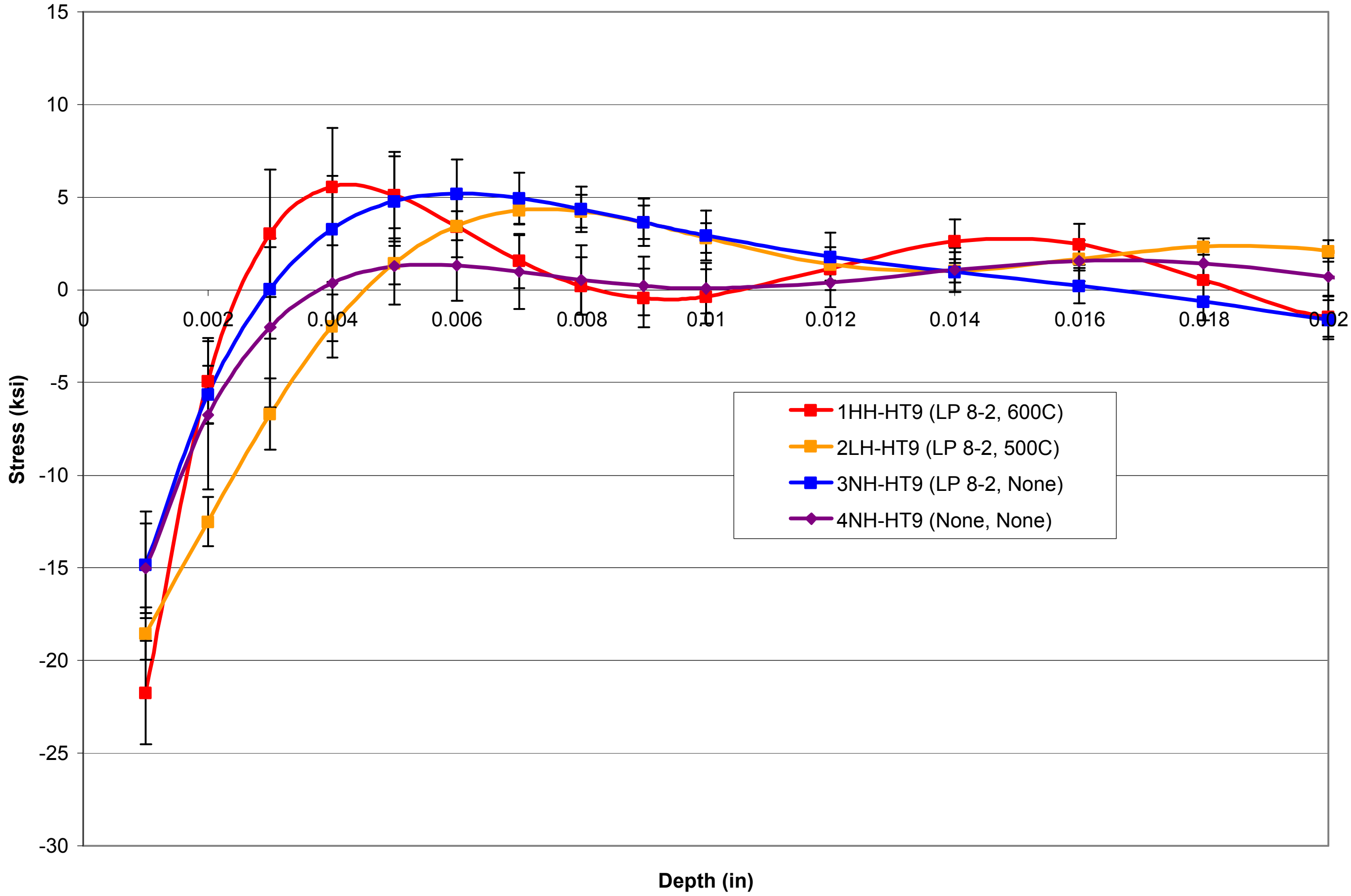


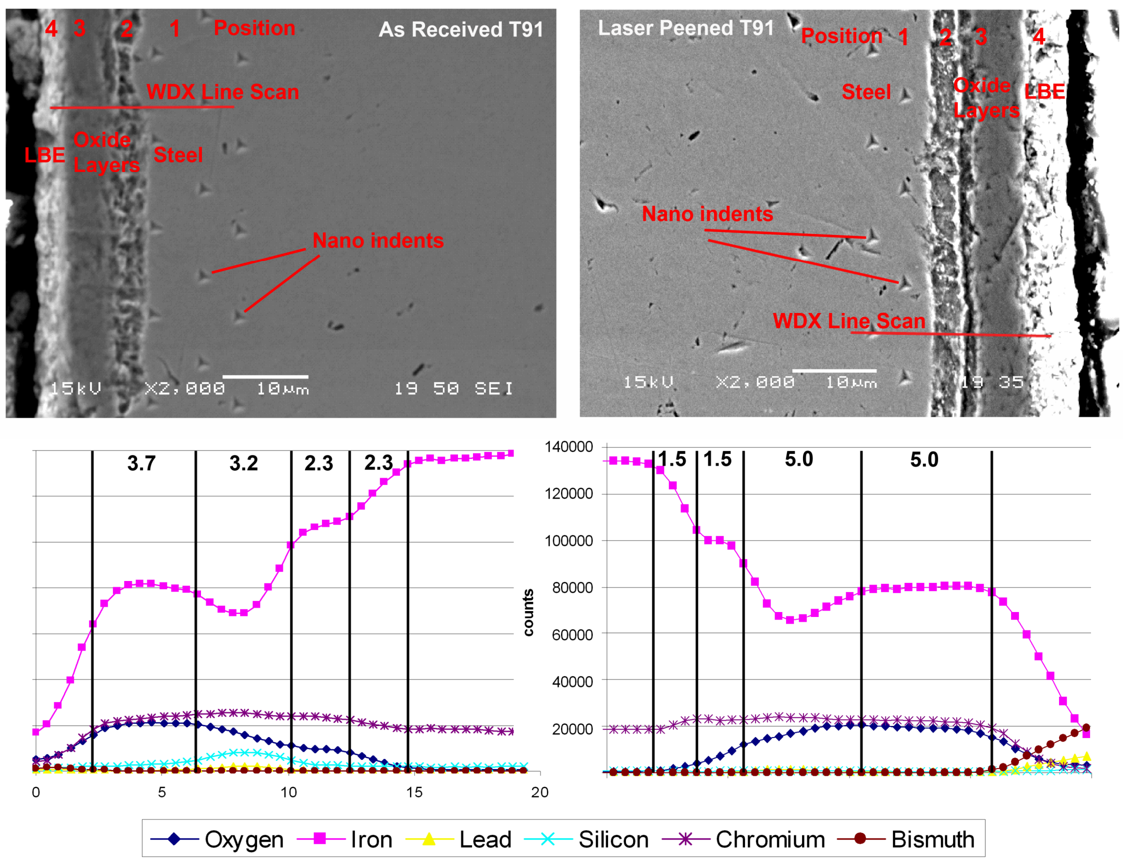
Fig. 11: Residual stress measurements for the 1mm thick HT9 coupons.

Further studies of thermal exposure effects are needed to quantify the magnitude and rate of thermal relaxation of the layer of compression induced by laser peening. We would also like to extrapolate that to higher temperatures (800 ^{o}C) corresponding to the case of Lead-cooled Fast Reactor operating conditions.

Corrosion Experiments

At the DELTA loop in LANL, SEM/EDX/WDX and nano indentation was performed on the sample cross section. The detailed results are presented in [6]. The SEM/EDX/WDX measurements on the Ferritic/Martensitic samples show no significant difference between laser peened and un-peened samples.

Fig. 12 presents the results of the performed measurements on the material T-91 after exposure to LBE. This can be seen in Fig. 12a and 12d.



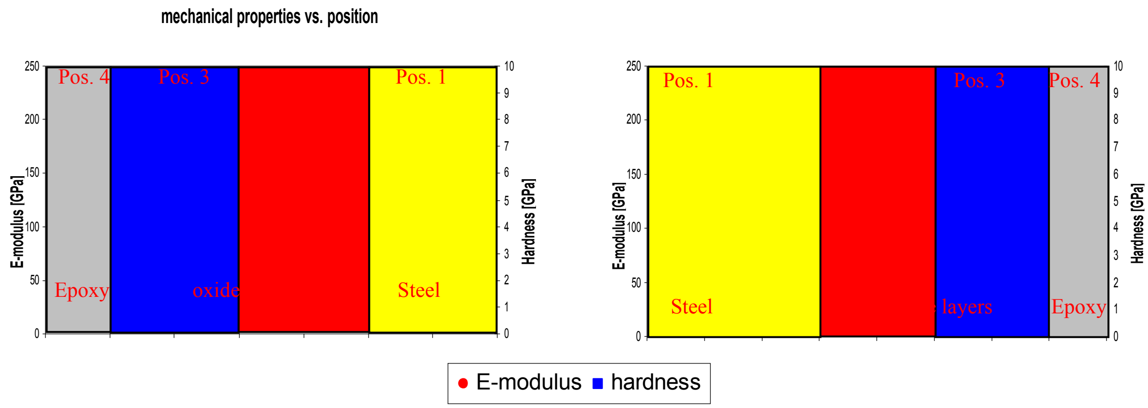


Fig. 12a and 12b present the SEM image and the corresponding WDX line scan of the as received T-91 material after 600 h exposure to LBE.

Fig. 12d and 12e present the SEM image and the corresponding WDX line scan of the laser peened T-91 material after 600 h exposure to LBE.

At both SEM images the nano indents are visible. The area where the nano indents were made (12c and 12f) is marked.

The appearance of the un-peened and laser peened material is very similar. Both materials formed a 5-6µm thick outer oxide layer and a ~3-4 µm thick oxide layer on their surface. The WDX measurements give more details of composition. There a diffusion zone, inner oxide layer, transition zone and outer oxide layer was identified. Also there both materials look very similar. The only difference might be that the laser peened material showed a slightly thicker outer oxide layer and thicker transition zone. But the thicker transition zone might be based on the gap between the two layers. This gap might be also the reason for the missing silicon signal in this area. On other line scans in other areas a clear Si signal was found.

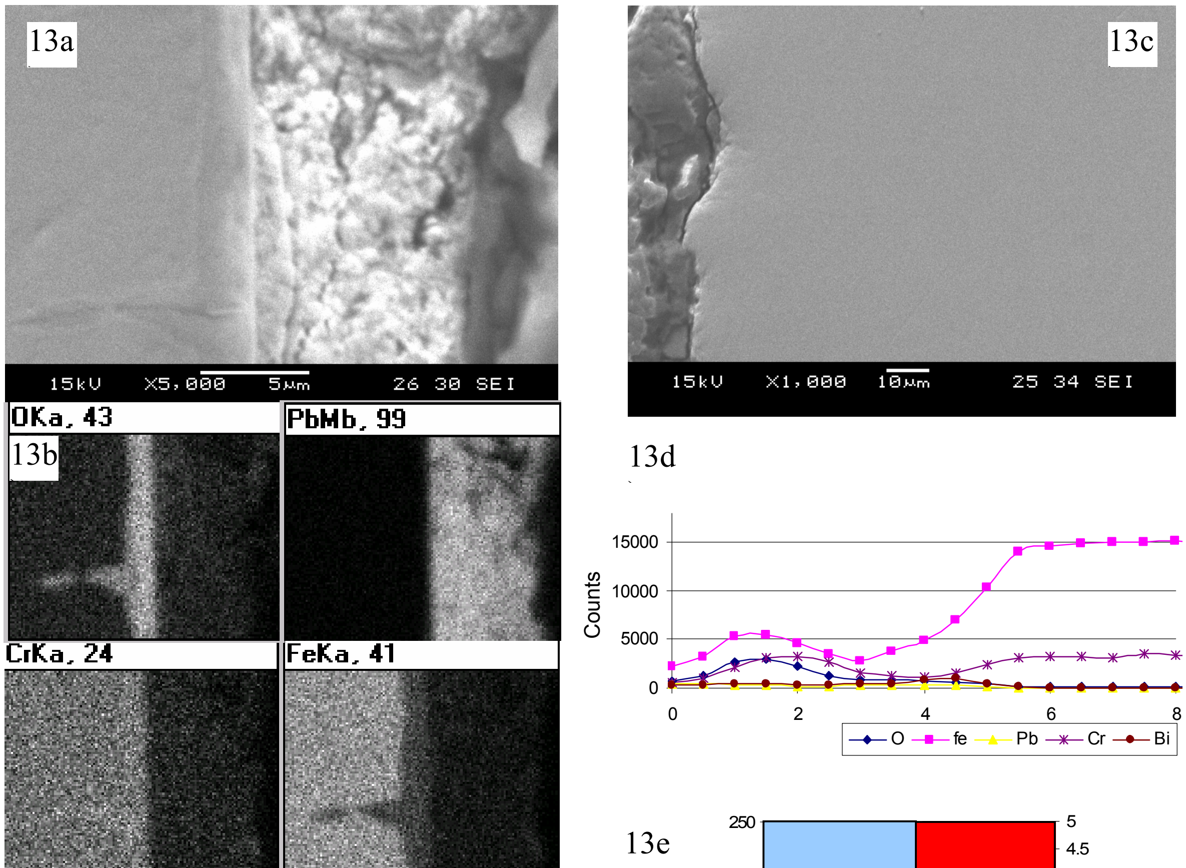
The nano indentation results show also a similar behavior of both samples. The E-modulus and hardness show a similar tendency. The steel has an e-modulus of about 200-160 GPa while the hardness is about 4 GPa. Both values are in the range of what was expected. In the layers the E-modulus drops 50-100 GPa while the hardness only slightly increases to max 8GPa. For a regular dense Fe Cr oxide a much higher hardness and E-modulus was expected. The only explanation is that the oxide layers have a high porosity which lowers these values. This is also in agreement with the literature [7]. There it was found that as the oxide grows thicker the density decreases (porosity increases) and therefore the hardness and E-modulus decreases. In this study, 8µm was the thickest oxide tested.

The relatively high deviation of the values can be understood by knowing that nano indentation is a localized method that strongly depends on surface effects. On each sample (laser peened and un-peened) 80 indents were made. But again, on F/M steels no significant difference between laser peened and un-peened was found. The other F/M materials (EP823 and HT-9) show a very similar behavior and also there no difference between the laser peened and un-peened materials was found.

The Austenitic material 316 L draws a different picture. It seems the laser peening protects the material from grain boundary oxidation as seen in Fig. 13. No continuous oxide layer was formed on the non-peened 316 L material as the oxidation mainly took place around and deep in grain boundaries.

The laser peened sample on the other hand showed some surface damage from the laser peening itself but no oxidation on grain boundaries and no continuous oxide layer. Fig. 13c presents a line scan of an un-peened sample in an oxidized area. 6e shows nano indentation results of the laser peened and un-peened bulk material. Indents in the oxide layers were not possible because the oxide layers were too thin and not continuous.

The second micrograph (Fig. 13b) corresponds to the 316 L unpeened sample. Note, that the orientation of the micrograph is reversed and the white region corresponding to the lead-bismuth layer now appears on the right hand side of the picture. A deep crack in the Fe-bulk is shown. The oxide layer penetrates the crack. The amount of Cr in the oxide layer is less than that observed in the laser peened region. Pb is present in the penetrated region.



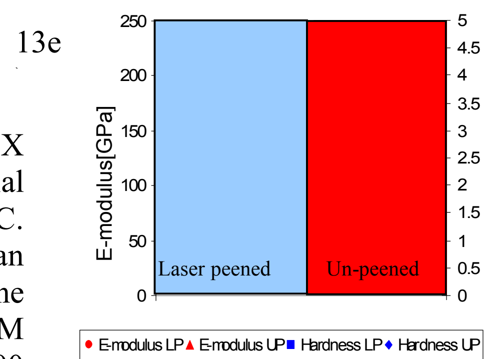


Figure 13a and 11b presents a SEM and EDX images of the un- peened 316 L material exposed to 400 h to a temperature of 535 °C. Figure 13d presents the line scan of an oxidized area on the un-peened material on the 600 h sample. Figure 13c presents the SEM images of the laser peened material after 400 h.Figure 13e shows the results of the nano indentation of the peened as well as the un-peened samples.

An annealing study [8] of high deformed (ECAPed) 316L showed that the work hardening effect stays in 316 L above an annealing temperature of 600 ºC and at 700 ºC the material loses the work hardening effect. Therefore it is assumed that the stress caused by laser peening stays in the material during the 535ºC/600h corrosion experiment. The effect of no oxidation in the grain boundaries at the laser peened sample can then be understood as well. It seems the compression stress on the surface makes it more difficult for the oxygen to diffuse inside the material.

LLNL EDS Results

EDS maps were performed on the limited amount of material available, see Fig. 14. The samples that participated in the DELTA loop corrosion test were sliced in two, a one half of the sample was sent back to LLNL for further investigations.

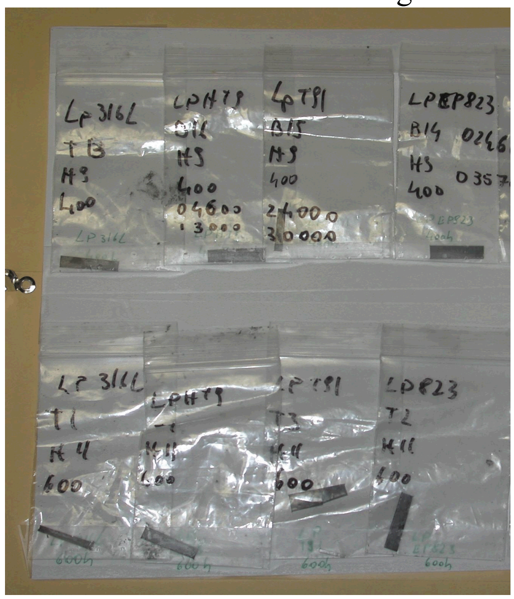


Fig. 14: Half-samples of 316 L, HT-9, T-91, and EP823 coupons sliced at LANL after participating in the lead-bismuth corrosion test lasting 400 and 600 h at 535 ^{o}C.

EDS maps of the laser peened region for the 316 L coupon are compared in Fig. 14 with those corresponding to the unpeened case. The figure shows that laser peening prevents LBE penetration in 316 L.

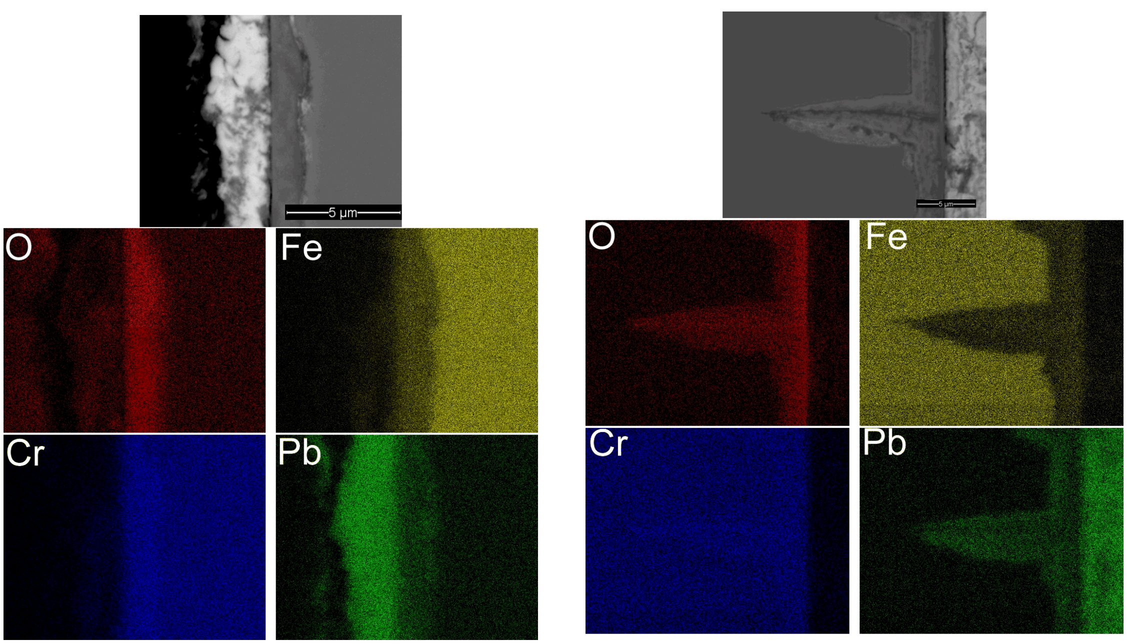


Fig. 15: Micrographs for 316 L sampes: a) peened and b) unpeened. Corresponding EDS maps for oxygen, iron, chromium and lead content in the sample.

These coupons were exposed to LBE at 535 ºC for 600 h. The first micrograph (Fig. 15a) shows results obtained for the 316 L peened sample. It shows a white region (~2.5 µm thick) corresponding to a lead-bismuth layer attached to the coupon, see also the strong Pb signal in the EDS map. This region is followed by a thick (~ 1.5 µm) Cr-rich and Fe-poor oxide layer that forms on the surface of the coupon during the corrosion experiment. In this region, the content of oxygen is also important. The presence of Fe in the bulk of the sample is shown in the Fe-EDS map.

Micrographs for HT-9 non-peened and laser peened samples are shown in Fig. 16. A white region corresponding to a lead-bismuth deposit covers the Cr-enriched oxide layer that lies facing the bulk of the sample (to the left of the picture). Laser peened and non-peened regions show the same chromium-oxide thicknesses. The oxide layer in the peened sample shows a smoothness that is not apparent in the non peened case. No LBE penetration is observed after 600 h exposure to LBE at 535 ºC.

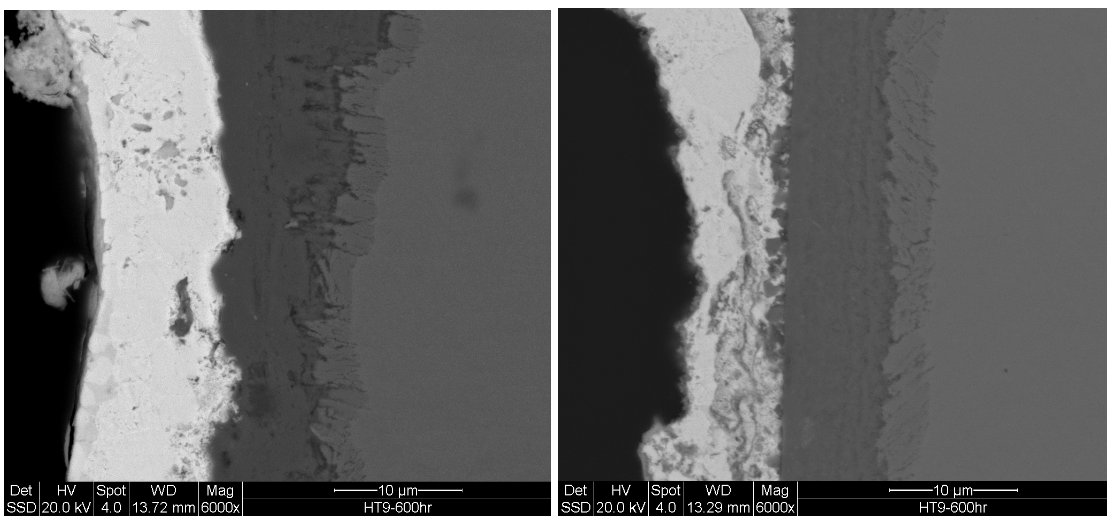


Fig. 16: HT-9 micrographs: a) non peened and b) peened samples.

Fig. 17 shows the EDS line map in HT-9 contains a Cr-enriched oxide layer ~7-8 µm in thickness (O in red, Cr in blue). This layer is depleted in Fe. A slightly less Cr enriched region extends into the bulk (to the left of the micrograph). The green line corresponds to the LBE layer shown in white in the micrograph.

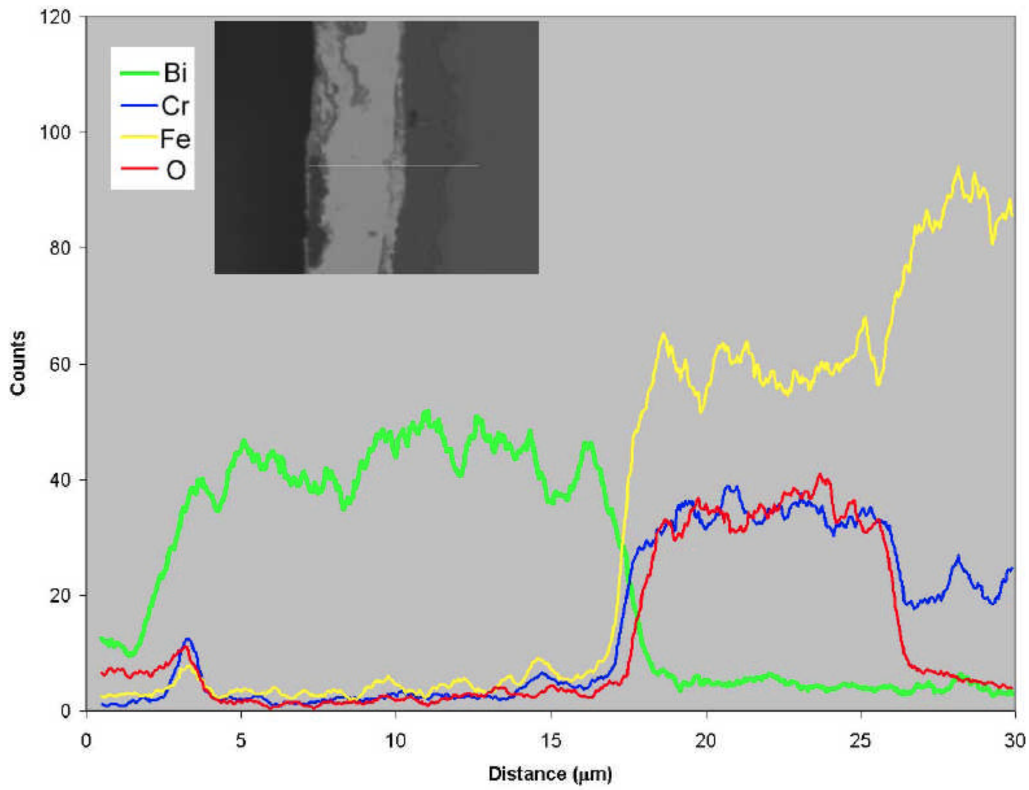


Fig. 17: EDS line in HT-9 and corresponding micrograph.

T-91 material was also investigated but is not reported here. No significant difference was found when comparing HT-9 and T-91 micrographs.

Future Experiments

There is great interest to have more laser-peened specimens for future irradiation testing. These samples could be laser peened to optimize laser parameters until the highest level of compressive stress is obtained. Also further thermal effects studies on RS relaxation could be done to differentiate behaviors that are not visible now due to the lack of experimental data. Differences between lead and lead-bismuth corrosive environments should also be investigated for the case of laser-peened surfaces. The effect of high doses of radiation, (~ 200 dpa) as those present at the cladding surface of the Gen-IV LFR, is still to be determined. A first set of LP T91, HT9, and 316L samples is participating in the STIP V irradiation campaign of the spallation neutron source in Paul Scherrer Institute (PSI), Switzerland.

Laser peening was done on ~1-mm thick flat specimens (slabs). Geometry optimization studies should show that the same performance is obtained in tubes intended for fuel cladding use. Welded pipes subjected to stress induced corrosion cracking and fatigue have already been laser-peened. The question is: What effects do the wall thickness and tube diameter have when considering laser peening as a solution to fatigue or SCC solution? Would laser peening be a cost effective treatment in the case of fuel-pin cladding?

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