Experimental report

17/02/2020

Proposal: **1-02-274**

Council: 4/2019

Title: USE OF NEUTRON DIFFRACTOMETRY FOR THE CHARACTERIZATION OF MICROSTRUCTURAL TRANSFORMATIONS FOLLOWING HIGHRATE DEFORMATION OF Ti6Al4V BY LSP

Research area: Engineering

This proposal is a new proposal

Main proposer: **Jose L. OCANA**

Experimental team: IGNACIO ANGULO
Jose L. OCANA
Francisco CORDOVILLA

Local contacts: Sandra CABEZA

Samples: Ti6Al4V

Instrument	Requested days	Allocated days	From	To
SALSA	5	3	20/01/2020	23/01/2020

Abstract:

The proposers have experimentally succeeded to introduce compressive residual stresses fields in Ti6Al4V with peak values in the range of 500 MPa and in material ranges beyond 1 mm depth that have conferred the treated specimens with clearly observable mechanical properties enhancement.
The proposers conducted a previous study with the double purpose of, first, to confirm the order of magnitude of the RSs fields induced in the material by LSP processes of a given intensity in rapport to the values measured by the ASTM E837-13 technique and, second, to confirm the relative stability of such RSs fields upon aggressive thermal cycles.
On the basis of these results, an additional degree of insight into the process is required by increasing the depth of exploration until at least few mm depth in order to observe the RSs recovery and the knowledge of the triaxial behaviour or the RSs fields. Neutron diffraction is hence the appropriate experimental approach to continue with. The correlation of experimental results with the available numerical simulation tools is critical in order to predict safely the mechanical behaviour of the treated samples along different directions.

USE OF NEUTRON DIFFRACTOMETRY FOR THE CHARACTERIZATION OF MICROSTRUCTURAL TRANSFORMATIONS FOLLOWING HIGH RATE DEFORMATION OF Ti6Al4V BY LSP

Dates: 20.01.20-23.01.2020

1. Initial aim and experimental plan

The samples under investigation are Ti6Al4V plates 50 x 50 x 7 mm^3 treated by Laser Shock Peening (LSP) in a region of 25 x 25 mm^2. Four samples will be investigated: TB-1, LB-1, TC-1 and LC-1. For Entry scans and maximum spatial resolution near the surface the collimators 0.6 x 0.6 x 0.6 mm^3 will be chosen. The experiment will complement previously developed studies about the characterization of residual stresses in the surface and near-to-the-surface regions of LSP samples.

2. Performed experiment

According to the disposition of treatment in the samples, the measurement of micro-deformations in the normal direction (perpendicular to the treated surface), required the configuration of the experimental setup for a reflexion disposition of the beam of neutron as shown in Fig. 1 to the left. While, the measurement in longitudinal and transversal direction required the transmission of the neutron beam throughout the analyzed sample.

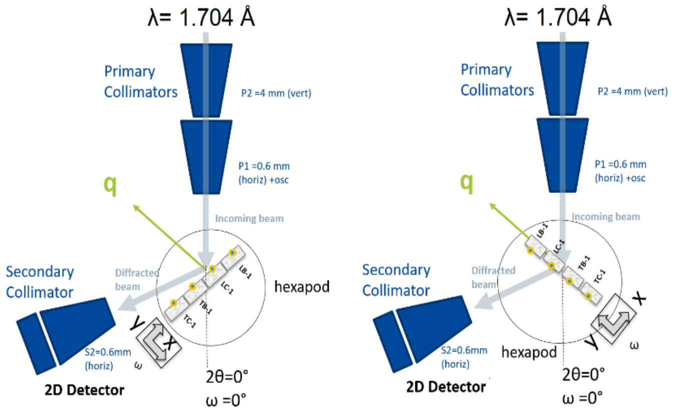

Figure 1: Experimental setup for normal direction in reflection position (left) and longitudinal and transversal directions in transmission disposition (right).

Due to the previously known specificities associated to the analysis of titanium and titanium alloys (as is the case of the working alloy, Ti6Al4V) by means of neutron diffraction (high background signal which has to be distinguished from the diffraction signal itself), the first works, after the accurate measuring and positioning of the samples, consisted in measurements in the bulk of the material of one of the samples to check the resolution of the expected diffraction peaks for the working material. This preliminary analysis showed that only for given orientation of the analyzed samples it was possible to detect the expected diffraction peaks corresponding to the crystallography of the material. A representative microstructure of the analyzed samples is shown in Fig. 2,3. Although a very slight orientation of the grains can be appreciated due to the manufacturing process of the samples, the degree of orientation keeps within a limit in which the microstructure can be considered as predominantly equi-axial with randomly oriented grains of α and β phases with average size. However, a combination of grain size of the particular plate together with texture

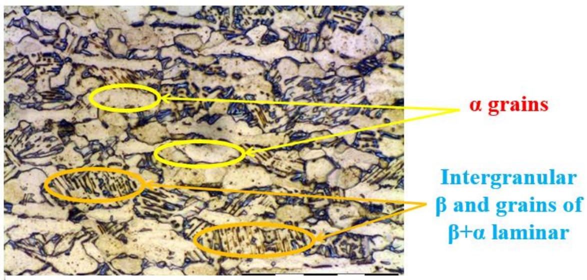

Figure 2: Illustrative microstructure of the samples

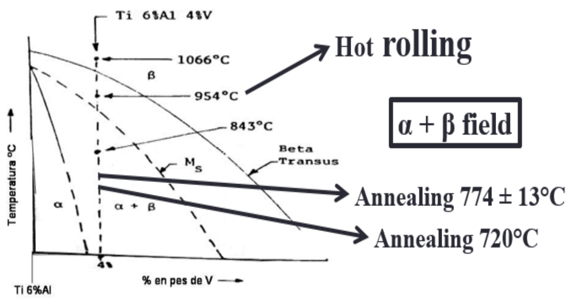

Figure 3: Region of interest of the phase diagram confirming that the microstructural phases are as characterized in the micrograph above.

is postulated as the possible reasons for peak extinctions in our tests. This was not foreseen from preliminary synchrotron tests due to the different material provider and the reduced penetration on those samples. Optical and electron microscopy of the base material is being addressed together with EBSD maps. A new plate substrate will be also purchase and characterize before next possible experiments.

3. Data reduction

In the normal direction it was not possible to obtain diffractograms for the crystalline planes corresponding to the Ti-103 as representative of macroscopic behavior of Ti6Al4V. A scan of the samples in angles around the normal direction ($\omega \pm 10°$) were perform to check the existence of diffraction peaks in a position close to the normal direction that could be representative of this one. Unfortunately no peaks were detected. Following a similar procedure to obtain the longitudinal direction, diffraction peaks were observed around 2θ = 45° (crystallographic plane 101), in a position 1° deviated from the nominal longitudinal direction, which is fully representative of the studied direction. A representative result for one of the analyzed samples is depict in Fig. 4a, where the diffraction peak is clearly in contrast with the background fitted by a Gaussian-kind profile. Proceeding in a similar way for the transversal direction, diffraction peaks were observed around 2θ = 42° (crystallographic plane 112), in a position 1.5° deviated from the nominal transversal direction, which is fully representative of the studied direction. A representative result for one of the analyzed samples is depict in Fig. 4b, where the diffraction peak is clearly in contrast with the background fitted by a Gaussian-kind profile.

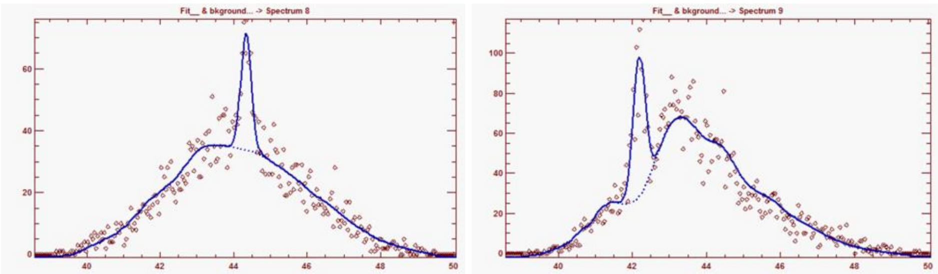

Figure 4: a) Illustrative diffraction peak in the longitudinal direction b) the transversal direction

4. Preliminary findings

Conventional residual stress calculation using neutron diffraction method usually requires three principal strain directions. However, the residual compressive stress field, induced by LSP processes along the applied load direction (normal direction in the present study) is usually negligible compared with the in-plane stress (Transverse and longitudinal directions). Therefore, the hypothesis of considering $\sigma_N = 0$ is adopted here *a priori* to calculate the nominal stresses along the longitudinal direction, σ_L, and the transverse direction, σ_T, An uncertainty for the stress along the normal direction, $\Delta\sigma_N$, is set equal to $\pm\, 0.2\ \max(|\sigma_L|, |\sigma_T|)$, leading to a corresponding reduced uncertainty in σ_L and σ_T. This confirms the relatively low sensitivity of the main stress field to be measured (along transverse and longitudinal directions) with respect to the normal stress, σ_N, suggesting the appropriateness of the alternative proposed method. For this first estimation, we also considered the bulk properties of the material for the Elastic modulus and Poission’s ratio, with future scope to implement it to each DEC of Ti-101 and Ti-112. However tendency will prevail and help us disclose in the short-term an overview of materials characterized.

Results for LB-1 type (LSP treated without post-thermal aging) and LC-1 type (LSP treated with post-thermal aging) are presented in Fig. 6. As expected the residual stress fields in the case of the aged sample experiences a significant reduction in comparison with the results from the non-aged sample. The good degree of agreement with the results of other techniques (i. e. Hole Drilling) for the measurement of residual stress in titanium alloys indicates the adequacy of the calculation method in similar depths. Despite this degree of agreement, the present results must be revised in accordance to the experimental facility calibration.

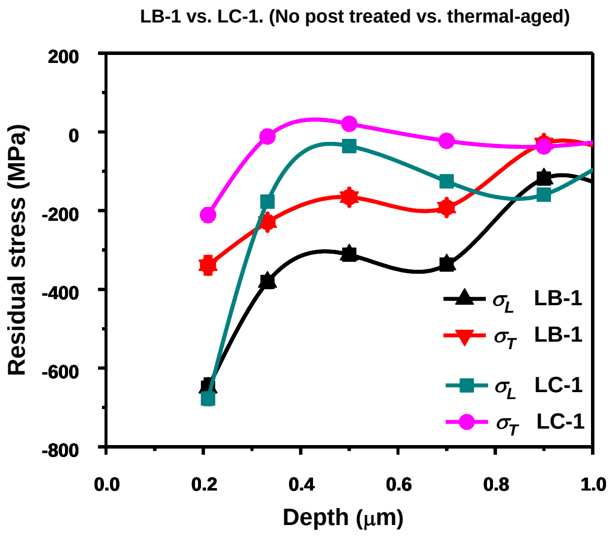

Figure 6: Residual stress curves in the longitudinal, σ_L and in the transversal direction, σ_T, for the sample LSP treated without thermal aging. We acknowledge that absolute values in MPa are not yet calibrated.

5. Preliminary Conclusions

The technique of neutron diffraction to measure residual stresses in a titanium alloy has been successfully applied to the experimental determination of residual stress fields in a typical industrial titanium alloy (Ti6Al4V).

It has been observe however, that even with a conventional equi-axial microstructure with grains of average size, only transmission measurements were possible. This suggests that the reflection phenomenon in the case of this material is conditioned by particular phenomena needing further theoretical-experimental study (i.e. microstructural characterization).

In spite of the lack of deformation measurements in the normal direction, the calculation of the residual stress of the analyzed points has been possible under the assumption that the stress in the normal direction is neglectable. This hypothesis constitutes a reasonable approach due to the wide knowledge about the plane stress state in samples submitted to LSP processes.

The residual stress results obtained by means of this methodology match, with a good degree of agreement, the results obtained by means of conventional mechanical techniques for the measurement of residual stresses fields in the working material.

The reported results needs to be further analyzed in connection to the experimental facility calibration inputs.