

Materials and methods

Sample preparation

Our study is carried out on the γ phase of TiAl alloys processed by SPS using standard methodologies already described elsewhere [33]. The starting materials are pre-alloyed gas-atomized powders of compositions $\text{Ti}_{48.7}\text{Al}_{47.3}\text{Cr}_{1.9}\text{Nb}_2$ (Crucible, Pittsburgh, USA, referred to as GE) processed at 1260°C [33], and $\text{Ti}_{50.1}\text{Al}_{47.9}\text{W}_{1.9}\text{B}_{0.1}$ (ATI, Pittsburgh, USA, referred to as IRIS) processed at 1350°C [34]. Cylindrical rods of 36 mm in diameter and 8 mm thick are obtained, from which specimens for mechanical tests are subsequently machined. Creep tests in tension are performed at 850°C under 150 MPa at a deformation rate of $6 \times 10^{-8} \text{ s}^{-1}$ until 1.5 % of deformation (see details in Ref. [6]). Specimens of TiAl deformed at 900°C are obtained by compression under vacuum of $3 \times 3 \times 7$ mm parallelepiped-shaped samples at a deformation rate of 10^{-4} s^{-1} until 2 % and 20 % of true plastic deformation. TEM thin foils are then extracted perpendicular to the creep or compression direction and prepared by electropolishing [35].

Electron tomography

The electron tomography investigations are performed with a JEOL 2010 TEM, using a GATAN double-tilt specimen holder. Micrographs are recorded under dark-field and weak-beam $\vec{g} \cdot n\vec{g}$ conditions ($n \sim 3-4$) giving high deviation to the Bragg's conditions and hence narrow dislocation lines, with steps of 1° from the minimum up to the maximum tilt value allowed by the sample holder (from -62° to $+62^\circ$), while excluding zone orientations where the signal/noise ratio is very low. The resulting stacks of $N \sim 100$ images are then processed using ImageJ's TomoJ plugin, for image registration [27, 36] and for 3D reconstruction [37]. Pre-alignment of the image stack is carried out using the cross-correlation function provided by the plugin. Then, main step of the registration procedure consists in aligning stacks of N images ($N \approx 100$) around a common tilt axis. However, because the images consist in orthonormal projections of the volume of the thin foils, the information of the (x, y, z) translational position of the physical tilt axis of the microscope is lost. Therefore, to achieve registration of the image stack in these conditions, a patch-tracking procedure has been implemented in TomoJ [27, 36]. It consists in defining regions of $p \times p$ pixels ($p \approx 15$), referred to as patches, on a first image (e.g. the image at tilt = 0). These patches are then scanned on the image in order to find characteristic features (landmarks) like intensity maxima. Then, correspondence of these landmarks is tracked in the next images in the stack. However, because the images are only approximately aligned at this stage, the number of images i in which patch correspondence is tracked has to be kept to low values at the beginning of the procedure ($i \approx 7$). These sets of aligned patches define thus $\approx 100-2000$ landmark chains of length i , for which the best alignment around circular arcs around the tilt axis is searched by computing shift, rotation, deformation shrinkage and deformation magnification corrections to the images. Then, this procedure is repeated by iteratively increasing i ($i: 7 \rightarrow N$), the alignment of the image stack improving at each step. This is therefore

an almost completely digital alignment procedure, in which “human” intervention occurs only in cases sub-stacks are individually aligned, but cannot be aligned with each other. In this situation, which can occur typically 2-3 times during the procedure, the sub-stacks are aligned manually using an image comparison tool provided by the TomoJ plugin. Finally, the image stack is considered as properly aligned when $i = N$. Then, the 3D reconstruction step, for which robust methods already exist, is carried out using a standard procedure (OS-SART algorithm [37], using the recommended parameters for number of iterations: 100, and for relaxation coefficient: 0.01). The resulting tomogram is then de-noised using a non-local means denoising algorithm [38, 39], and the final 3D reconstruction is visualized using the freeware Tomviz (<https://tomviz.org>).

References

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