Rotational defects as stabilising elements in SPD materials

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Abstract. In this paper the focus is laid on the thermal stability of nanocrystalline materials obtained by severe plastic deformation. TEM investigations show that rotational defects act as stabilising elements in grains as well as at grain boundaries.

1. Introduction
The thermal stability of ultrafine grained (ufg) or nanocrystalline (nc) materials is an important issue and thus of technological interest. Grain growth occurs in polycrystalline materials to reduce the total energy of the system introduced by the grain boundary (GB) area. Therefore, the high density of interfaces in ufg or nc materials is likely to provide a significant driving force for grain growth. Systematic studies of grain growth in nc Pd at room temperature were carried out [1]. It was shown that the thermal stability of nc materials can be improved either by the addition of solutes [2] or by severe plastic deformation (SPD) [3]. In the present study we summarize our comprehensive findings regarding the enhancement of thermal stability by deformation, based on quantitative strain analyses using geometric phase analysis (GPA) [4].

2. Experimental
The material investigated was nc Pd produced by inert gas condensation (IGC) [1, 5]. The material was deformed either by cold rolling [6] or high-pressure torsion (HPT) [3]. Electron transparent samples were prepared by either electro-polishing or ion-milling and subsequently investigated using state-of-the-art transmission electron microscopes, see references [6-8]. The GPA method developed by Hýtch and co-workers [4] was used to measure and map strain fields in high-resolution TEM images. The in-plane strain tensor components, ε_{ij}, and the in-plane rigid-body rotation, ω_{ij}, were calculated using a commercial version, GPA Phase 2.0 (HREM Research).

3. Results and Discussion
Microstructural investigations of cold-rolled nc Pd showed that room temperature grain growth occurred [6]. However, it was found (Fig.1) that some regions within the coarse grains remained nanocrystalline.
A closer inspection of such stable nanocrystalline regions showed that they often contain twins and deformation debris (Fig. 2a). This observation suggests that deformation, which is a heterogeneous process, provided elements stopping such grains from growing. To investigate this hypothesis further, quantitative strain analyses were carried out using GPA. Fig. 2b shows a colour-coded map of the rigid-body rotation $\omega_{xy}$ of the Pd nanograin. The profile of the rigid-body rotation averaged over the boxed area in the central matrix segment of the nanograin is displayed in Fig. 2c. This profile shows unambiguously the existence of rotational gradients. Two turning points indicated by arrows are observed in the profile. Thus, the top and bottom parts of the Pd grain are rotationally distorted with respect to each other. Since such structures have been observed frequently and are thermally stable over long periods (> 1 year) at room temperature, it is concluded that such rotational defects represent a mechanical equilibrium which act as stabilising elements against grain growth. The strain energy stored in such a deformed Pd grain was estimated to be about 4 J g$^{-1}$ [7].
Moreover, it may be speculated that such rotational defects manifested in the grain interiors would elucidate the process of grain refinement, which is an essential part of SPD processing [9]. Interestingly, rotational defects were also observed at grain boundaries. Fig. 3a shows a high-resolution micrograph of a part of another Pd grain processed by HPT that was also found to be stabilised against room temperature grain growth [3, 8]. The microstructure shown in Fig. 3a contains a triple junction, which consists of two intersecting Σ3 twin boundaries with a Σ9 boundary. The three intersecting boundaries result in a visible net translation (closing failure) of a<sub>n</sub>/4 <211> (see Burgers circuit in Fig. 2a). Thus the core of the triple junction exhibits dislocation character [8]. Moreover, the Σ9 grain boundary forms a quadruple junction further down with three intersecting Σ3 twin boundaries. It has been shown that triple and quadruple junctions have a significant influence on grain growth [10-12]. The question is: Why is this configuration stable? To answer this question, a detailed inspection of this configuration has been carried out using GPA (Fig. 3b,c). The strain analysis of the rigid-body rotation \( \omega_{xy} \) is depicted as a colour-coded map in Fig. 3b. The rigid-body rotation...
rotation was quantified along the $\Sigma 9$ boundary in the form of a profile starting at the triple junction, ending near the quadruple junction and averaged over the boxed area. Two gradients of opposite sign emerging at the triple point and the quadruple junction are observed in Fig. 3c. A comparison with the HRTEM image (Fig. 3a) discloses that the atomic columns are distorted where the gradients merge. The hashed line indicates the average grain misorientation of a $\Sigma 9$ boundary having no defects. This information was obtained from a MD simulated $\Sigma 9$ boundary, having no rotational defects, and subsequently quantified by GPA (Fig. 3d). The comparison of Fig. 3c with Fig. 3d shows that a disclination emerging from the triple point along the $\Sigma 9$ grain boundary is balanced by a disclination of opposite sign emerging from the quadruple junction. Thus such a dipole configuration represents a local mechanical equilibrium which inhibits the annihilation of the $\Sigma 9$ grain boundary. It is concluded that the presence of this disclination dipole is essential for the stability of the microstructure.

Fig. 3: (a) Lattice image (taken from reference [8]) showing part of a heavily deformed nc Pd grain containing a triple and quadruple junction, respectively. (b) Corresponding strain map (rigid-body rotation). (c) Strain profile measured along the boxed area. (d) Strain profile of a simulated $\Sigma 9$ boundary used for comparison.

4. Conclusions
Quantitative strain analyses of severely deformed microstructures of nanocrystalline Pd showed unambiguously the presence of rotational defects in grain interiors as well as at grain boundaries. Since such microstructures were found to remain stable for a long time at room temperature, it is concluded that rotational defects are stabilising elements inhibiting grain growth.

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References
[1] Ames M et al 2008 Acta Mater. 56 4255
[2] Krill C E, Erhardt H and Birringer R 2005 Z. Metallkd 96 1134
[3] Ivanisenko Y et al 2009 Acta Mater. 57 3391
[4] Hýtch M J, Snoeck E, Kilaas R 1998 Ultramicroscopy 74 131
[5] Birringer R et al 1984 Phys. Lett. A 102 365
[6] Rösner H, Markmann J and Weissmüller J 2004 Phil. Mag. Lett. 84 321
[7] Rösner H et al 2010 Acta Mater. 58 2610
[8] Rösner H et al 2011 Acta Mater. 59 7380
[9] Tóth L S et al 2010 Acta Mater. 58 1782
[10] Gottstein G, Shvindlerman L S, Zhao, B 2010 Scripta Mater. 62 914
[11] Zhao B, Gottstein G, Shvindlerman L S 2011 Acta Mater. 59 3510
[12] King A H 2010 Scripta Mater. 62 889