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Generalized scaling of misorientation angle distributions at meso-scale in deformed materials

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Scaling behaviour has been observed at mesoscopic level irrespective of crystal structure, type of boundary and operative micro-mechanisms like slip and twinning. The presence of scaling at the meso-scale accompanied with that at the nano-scale clearly demonstrates the intrinsic spanning for different deformation processes and a true universal nature of scaling. The origin of a ½ power law in deformation of crystalline materials in terms of misorientation proportional to square root of strain is attributed to importance of interfaces in deformation processes. It is proposed that materials existing in three dimensional Euclidean spaces accommodate plastic deformation by one dimensional dislocations and their interaction with two dimensional interfaces at different length scales. This gives rise to a ½ power law scaling in materials. This intrinsic relationship can be incorporated in crystal plasticity models that aim to span different length and time scales to predict the deformation response of crystalline materials accurately.

A complete understanding of the plastic deformation behaviour of metals and alloys has been a great challenge for the scientific community engaged in metal physics due to the processes occurring over wide length and time scales[1]. Studies ranging from micro-scale (molecular and dislocation dynamics at atomic level) and meso-scale (visco-plastic self-consistent models and crystal plasticity finite element models at the grain level) to continuum scale (finite element methods at structural level) as well as multi-length scale approaches have been employed to understand plasticity[2–4]. However, a common law valid over these length scales is still elusive. Allometric scaling laws which link the size of an organism with its shape, behaviour and physiology are known to be valid for all organisms covering as high as twenty one orders of magnitude of length scales (sizes)[5]. Similar issues are encountered urban planning with respect to urban infrastructure and socio-economic output[6]. It is therefore, worthwhile to study whether such a law exists for different processes occurring during deformation of crystalline materials over the pertinent spatiotemporal space.

It is well established that dislocations act as the carriers of plasticity and their gradual interaction with each other leads to work hardening that aids in avoiding early fracture in crystalline materials. In addition to the evolution of dislocation substructure and consequent work hardening, large strain deformation of metals and alloys results in gradual reorientation of the crystallites that leads to a preferred crystallographic orientation or texture[7,8]. A number of investigations are dedicated to examine the plastic deformation of materials at different length scales; however, a unified approach that can be extended to all the length scales is still missing. In classical physics, statistical mechanics has been employed to address various issues spanning over a large length and time scales[9]. If plastic deformation is considered as a far from equilibrium thermodynamic process, the concept of statistical mechanics can be extended to develop the understanding of various deformation processes[10]. The presence of scaling of misorientation was attributed to stochastic nature of dislocations[10]. A few detailed studies on microstructural evolution in deformed face centre cubic (FCC) metals and alloys using Transmission Electron Microscope (TEM) has shown a scaling behaviour for misorientation[11,12]. These studies have revealed that the average misorientation increases with increase in strain ($\theta_{avg} \propto \sqrt{\varepsilon}$), however, the modified distribution showed a scaling behaviour. In addition to misorientation, scaling behaviour was also observed for the cell size in deformed FCC metals[13,14]. This constraint, as manifested by TEM observations is applicable to the theories trying to address the problem of plastic deformation from the bottom up approach like dislocation dynamics simulations. No such constraints are imposed on the approaches dealing with the meso and macro scale aspects of plasticity. Therefore, there is a need for a unified theory that can explain the plastic deformation behaviour inclusive of all length scales.

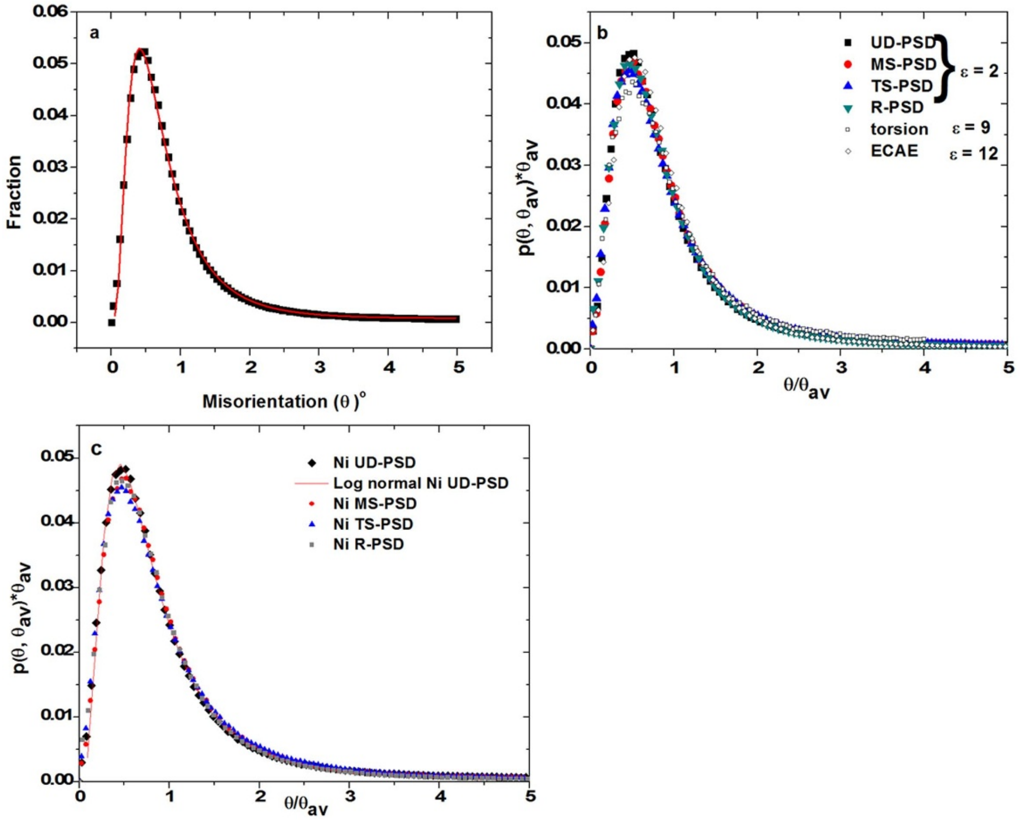

Figure 1 | (a). Misorientation distribution for UD-PSD sample and Modified misorientation distribution with respect to the average misorientation for nickel. (b). Samples deformed by different strain paths and (c). Comparison of the modified distributions with a log-normal distribution.

The evolution of Scanning Electron Microscopy based Electron Back Scatter Diffraction (EBSD) technique provides an experimental way to bridge the gap between the micro/nano length scale on one hand and meso/macro scale on other[15]. With the advent of microscopes with Field Emission source, EBSD can provide truly nanoscale resolution, and an angular resolution of ~1° can be obtained on a routine basis. Visco-plastic self-consistent simulations and crystal plasticity finite element simulations use the data obtained from EBSD to fine tune the model parameters in terms of slip and twin activity[16,17]. Recently, the evidence of spatial correlation in high angle misorientation distribution obtained from EBSD microstructures has been demonstrated[18]. The scale free intergranular spatial correlations were attributed to the long range internal stresses arising due to geometrically necessary dislocations near the boundary that help in maintaining strain compatibility between the neighbouring grains. This observation puts an additional constraint on the continuum simulations so as to reproduce the spatial correlations observed experimentally.

Results

We now present our data on misorientation distribution [$p(\theta)$] obtained from EBSD for Ni samples subjected to different forms of plane strain deformation, shear deformation as in torsion and severe plastic deformation by Equal Channel Angular Extrusion (ECAE). The different modes of plane strain deformation by rolling comprise of unidirectional (UD-PSD), reverse (180° change in rolling direction, R-PSD), multi-step (90° change in rolling direction in each step, MS-PSD) and two-step (90° change in rolling direction at half the original strain, TS-PSD) routes. Earlier investigations by the authors have addressed issues pertaining to evolution of microstructure and texture in nickel as a function of different strain paths[19–21]. Thus, the aforementioned processes cover a wide range of strain, strain rates and strain paths including a severe plastic deformation process[22–24]. The misorientation distribution obtained from all the samples was found to be log-normal in nature. The misorientation distribution for nickel UD-PSD sample with log-normal fitting (red curve) is shown as a representative in Fig. 1a. The distribution shows a good match with a log-normal distribution. It has been found that the probability of a boundary selected at random associated with misorientation angle between θ and $\theta + d\theta$ (given the average misorientation angle is θ_{av}); that is the modified θ distribution in terms of $\theta_{av} * p(\theta, \theta_{av})$, shows a universal behaviour (Fig. 1b). A good match is obtained with the log-normal distribution for all the Ni samples subjected to different strain paths (Fig. 1c). Similar scaling behaviour exists for body centre cubic (BCC) Interstitial Free steel (Fig. 2a) subjected to different passes of ECAE deformation and hexagonal close packed (HCP) titanium (Fig. 2b) subjected to quasi-static compression. The experimental details of all the samples studied in the present investigation are given in Table 1. Complete investigation of microstructural evolution and texture analysis is documented in details elsewhere[25,26]. The occurrence of scaling irrespective of pixel size and shape indicates that it is not associated with noise associated with data capturing in EBSD. The occurrence of scaling in HCP titanium is particularly interesting as in addition to normal dislocation slip, substantial deformation twinning (extension $10\bar{1}2$ and contraction $2\bar{1}\bar{1}2$) is active in some samples. The occurrence of scaling of misorientation in the presence of twinning is been reported for the first time in this investigation.

In order to ascertain that scaling behaviour of misorientation in EBSD is not an artefact of the measuring technique, we examined the effect of strain on the misorientation evolution[27]. The misorientation build up in terms of grain reference misorientation was monitored in a cluster of grains in an oligocrystalline nickel sample with deformation (Fig. 3a). The effect of strain on misorientation distribution as seen in Fig. 3b indicates increase in the average misorientation with the square root of strain (Fig. 3c) as reported by TEM observa-

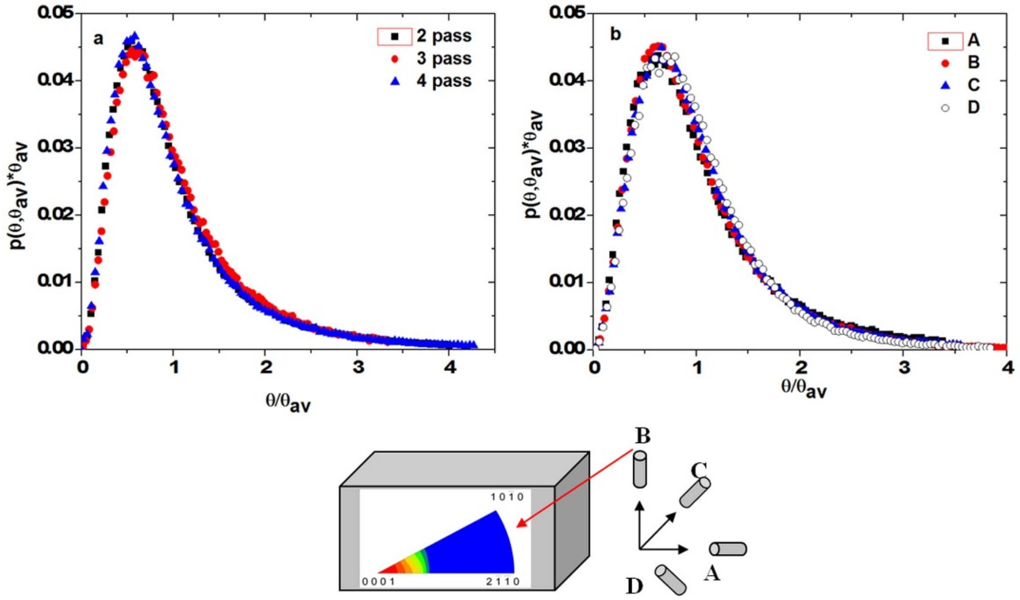

Figure 2 | Modified misorientation distributions scaled with respect to the average misorientation for (a). Interstitial Free steel subjected to different Equal Channel Extrusion passes and (b). Titanium samples with different texture, deformed to strain of 0.35 in compression at room temperature.

tions[11,12]. This clearly shows that once a proper step size is selected for EBSD in accordance with the Humphrey's criterion[28], the scaling behaviour can be reproduced successfully.

Discussion

Earlier investigation by Hughes et al.[11,12] shows better scaling behaviour for Incidental Dislocation Boundaries (IDBs) compared to the boundaries formed from the geometrically necessary dislocations that is the geometrically necessary boundaries (GNBs). No scaling behaviour was observed when IDBs and GNBs were considered together. Hähner et al.[13] attributed the fractal nature of dislocation substructure to the noise generated due to dislocation movement and self organized criticality at multiple length scales during deformation. Other researchers have attributed it to multi-slip[29] and orientation diffusion in Euler space[30]. Recently, Chen et al.[31,32] proposed a minimum continuum model for meso-scale plasticity in 2D and 3D which accounted for cellular microstructures in deformed crystals. However, in the present investigation, scaling behaviour is observed for the combination of IDBs and GNBs. This can be attributed to the multiplicative nature of the various dislocation processes operative during plastic deformation.

The occurrence of similar scaling behaviour at nano-, micro- or meso-scopic length scales has other important consequences. Material behaviour tends to show a scaling with power law exponent of ½. For example, misorientation and strain, $\theta \propto \sqrt{\varepsilon}$; shear stress and dislocation density $\tau \propto \sqrt{\rho}$, yield stress and grain size[33,34] $\sigma \propto 1/\sqrt{D}$ or grain growth kinetics[35] $R \propto D^2$. It is to be mentioned here that grain growth is analogous to thinning (reduction in population of dislocations and hence shows a reciprocal of ½ that is 2 as power law exponent). Thus unlike living organisms in Euclidean space that exist in four dimensions (3 Euclidean and one fractal), materials tend to show a decrease in spatial dimension. This could very well be attributed to the fact that most of the activities occurring in materials are governed by processes taking place at the interfaces (atomistic planes containing dislocations, IDB, GNB, low and high angle grain boundaries) which are essentially two dimensional. This is actually inherent to plastic deformation as dislocations that comprise the smallest constituent of plastic deformation have a two dimensional plane separating a region of compressive and tensile stresses. A schematic showing the importance of interfaces in deformation behaviour of crystalline materials is shown in Fig. 4. Thus, the presence of scaling of misorientation at multiple length scales is apparent.

Table 1 | Details of the various samples studied in the present investigation. The different modes of processing are uni-directional plane strain deformation (UD-PSD), multi-step (MS-PSD), two step (TS-PSD), reverse plane strain deformation (R-PSD), torsion, and equal channel angular extrusion (ECAE) by route B_c that involves 90 degree rotation after each pass

Serial No.	Sample	Step size (nm)	Pixel shape	Comment
1	Ni UD-PSD ($\varepsilon = 2$)	100	hexagonal	-
2	Ni MS-PSD ($\varepsilon = 2$)	100	hexagonal	-
3	Ni TS-PSD ($\varepsilon = 2$)	100	hexagonal	-
4	Ni R-PSD ($\varepsilon = 2$)	100	hexagonal	-
5	Ni torsion ($\varepsilon = 9$)	50	hexagonal	-
6	Ni ECAE 12 pass ($\varepsilon = 12$)	50	hexagonal	-
7	IF steel ECAE 1 pass ($\varepsilon = 1$)	50	square	-
8	IF steel ECAE 2 pass ($\varepsilon = 2$)	50	square	-
9	IF steel ECAE 3 pass ($\varepsilon = 3$)	50	square	-
10	Ti A ($\varepsilon \sim 0.35$)	1000	square	Extensive extension twins + substantial contraction twins
11	Ti B ($\varepsilon \sim 0.35$)	1000	square	Substantial contraction twins + extension twins
12	Ti C ($\varepsilon \sim 0.35$)	1000	square	Extensive extension twins + substantial contraction twins
13	Ti D ($\varepsilon \sim 0.35$)	1000	square	Extensive extension twins + substantial contraction twins

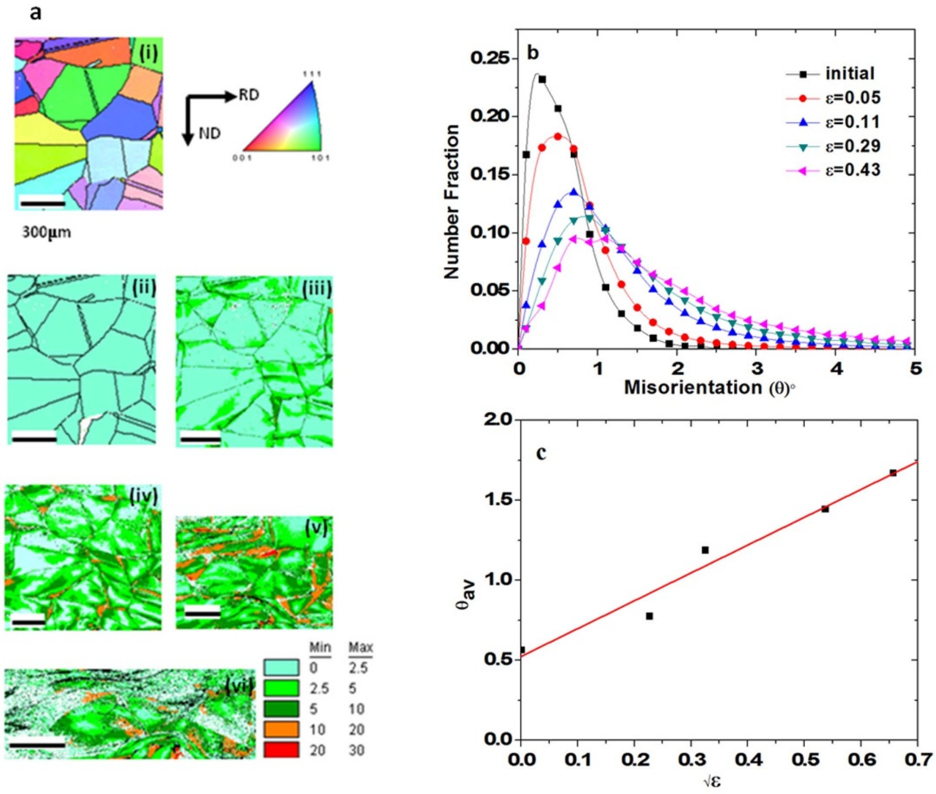

Figure 3 | (a). (i) Orientation Map in the as received condition and grain reference orientation deviation map of the (ii) initial and deformed nickel oligocrystal to strain of (iii) 0.05 (iv) 0.1 (v) 0.29 and (vi) 0.43 (b). Misorientation distribution for the same sample as a function of strain and (c). Evolution of misorientation distribution with strain.

The observation and analyses presented above leads to the conclusion that the scaling behaviour is independent of not only material properties like stacking fault energy, solute content etc. and process parameters like temperature, strain, strain rate, strain path etc. but also the crystal structure and presence of additional deformation mechanism like twinning[36]. We have shown that the scaling of misorientation distribution is inherent of any deformation process irrespective of the ability of the material to form cell structure and is applicable to both IDBs and GNBs at the meso-scale. This clearly indicates that the scaling behaviour is truly universal. Unlike in the TEM, the scaling of misorientation arising in EBSD may not be only from the IDBs or GNBs but also from the stray dislocations trapped within the grains. Therefore, a better model is necessary to explain the scaling behaviour than the one based on the Incidental and Geometric Dislocation Boundaries.

In addition to being faster and easier, however, less accurate (angular resolution $\sim 1^\circ$) than TEM, EBSD provides additional information about the long range and short range misorientation parameter like Kernel Average Misorientation (TSL-OIM EBSD User Manual for OIM Analysis version 5.2). Recently, Zhong et al.[37] had shown that Kernel Average Misorientation (KAM) corresponds to the deformation microstructure and can be used to study the evolution of substructure during deformation. The KAM image of the nickel sample (few representative grains) subjected to unidirectional plane strain deformation (UD-PSD) is shown in Fig. 5a along with the pattern quality map. The similarity between the two is quite astonishing and the regions of low Image Quality that essentially indicate boundaries, correspond to high KAM. Therefore, one would believe that a cut-off value for KAM could be used to distinguish IDBs and GNBs in future. However, no scaling behaviour was observed for KAM distribution (Fig. 5b) of the deformed samples at least with the average KAM as a scaling parameter. The study of the distribution of the secondary misorientation parameters may shed new insights in understanding plasticity in general and strain-gradient plasticity in particular[38].

The occurrence of scaling behaviour in EBSD data puts an additional constraint on the various deformation models like the mesoscopic self consistent models and the continuum crystal plasticity finite element methods. Already, Lebensohn et al.[39] have developed the "second order approximation" in viscoplastic self-consistent formulation that calculates the average field fluctuations inside the grains of a polycrystal by calculating the second order moments of stress. The presence of scaling of misorientation can be incorporated in the second order formulation. Similarly for the various CPFEM models using multiple elements per grain[40], it is mandatory to satisfy the scaling behaviour of low angle misorientation that has been demonstrated in the present investigation.

In conclusion, the evidence of scaling of misorientation from EBSD data in FCC, BCC and HCP metals for a combination of IDBs and GNBs indicate the stochastic nature of dislocation plasticity in these materials irrespective of the crystal structure and dominant deformation mechanisms. The evidence of scaling from EBSD data puts an additional constraint on meso-scale simulations for microstructure and texture evolution. Better prediction of crystallographic texture is expected by incorporating the scaling behaviour of misorientation in the meso and continuum models. However, the exact reasons for scaling behaviour of misorientation irrespective of character of the boundary (IDB or GNB), crystal structure, dominant deformation mechanism and various processing parameters like strain rate, strain, temperature still remain an open issue. The presence of a ½ power law scaling in crystalline materials at different length scales indicate that the interaction of dislocations with two

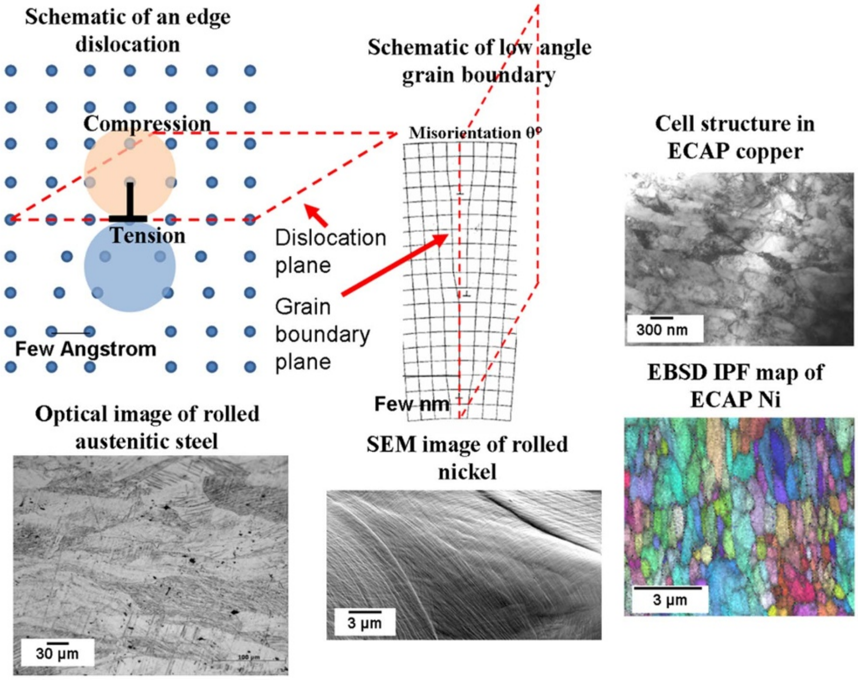

Figure 4 | The contribution of interfaces in single phase materials over different length scales. Two dimensional interfaces be it atomic plane separating tensile and compressively stressed lattice above and below a dislocation or a low angle grain boundary formed by array of dislocations or IDBs and GNBs or low angle grain boundary and high angle grain boundary play an important role in deformation behaviour of crystalline materials.

dimensional atomic planes or different kind of boundaries plays an important role in deciding deformation behaviour of crystalline materials.

Methods

Polycrystalline nickel samples were subjected to plane strain deformation (PSD) by cold rolling using a two-high rolling mill to 90% resuction at room temperature. Four distinct strain path comprising of uni-directional plane strain deformation (UD-PSD), multi-step (MS-PSD, intermittent change in rolling direction by 90 degree), two step (TS-PSD, change in rolling direction by 90 degree after 50% true strain deformation) and reverse planes strain deformation (R-PSD) comprising of change of rolling direction by 180 degree. Shear deformation was carried out using free end torsion test on cylindrical sample and equal channel angular extrusion was carried out on billets of size 10 mm by 10 mm by 100 mm using an indigenously designed 90 degree die at room temperature. Torsion test was carried out till the sample fractured at a strain of 9 while ECAE was carried out till 12 passes using Route B_c that comprised of rotation of the sample by 90 degrees after each pass to provide an uniform microstructure. Interstitial Free steel (IF steel) samples were also subjected to ECAE to 3 passes at room temperature using the same die and same route. Titanium samples were obtained in different orientations along the longitudinal, transverse and normal

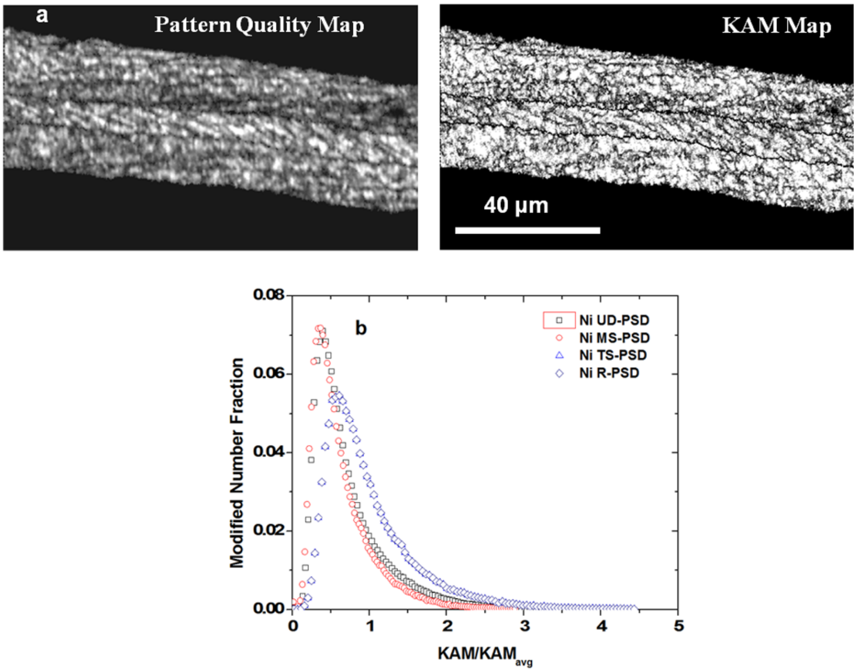

Figure 5 | (a). Pattern Quality image for a region of the Uni-directional plane strain deformed sample (left) and corresponding Kernel Average Misorientation image (right) and (b). Modified distribution for Kernel Average Misorientation for the differently plane strain deformed samples.

direction from a rolled block of titanium with a strong basal texture to obtain samples with different initial texture. These samples were then subjected to high strain rate compression using a Split Hopkinson Pressure Bar setup to a strain of 0.35. The rolled nickel samples were metallographically prepared and electro-polished for EBSD observation on the mid transverse plane. The ECAE processed IF steel samples were metallographically prepared and electro-polished to obtain microstructure on the transverse plane. Similarly, titanium samples were prepared to observe the microstructure at the mid-thickness of the plane containing the compression direction. EBSD was performed on a Field Emission Scanning Electron Microscope (FE-SEM) Sirion with TSL-OIM Data Collection software version 5.2. Data analysis was carried out using TSL-OIM Data Analysis software version 5.2.

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Author contributions

S.S. and N.G. conceived and designed the experiments. N.G. performed the experiments and data analysis in consultation with S.S. S.S. and N.G. wrote the manuscript.

Additional information

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