Spatial distribution of defects in ultra fine grained copper prepared by high pressure torsion

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Abstract. Bulk materials with ultra fine grain structure can be fabricated by severe plastic deformation. Among variety of techniques based on severe plastic deformation high pressure torsion is the most efficient method for grain refinement down to nano-scale. In torsion deformation the strain distribution across the sample is non-uniform and increases with increasing radial distance from the centre of the sample corresponding to the axis of torsional straining. Due to this reason it is very important to examine homogeneity of ultra fine grained structure of samples prepared by high pressure torsion. In the present work positron annihilation spectroscopy was employed for mapping of spatial distribution of defects in ultra fine grained copper prepared by high pressure torsion. Spatial distribution of defects was examined by means of (i) Doppler broadening using $S$ parameter for mapping of defect density and (ii) positron lifetime spectroscopy. Spatially resolved positron annihilation studies were combined with mapping by microhardness testing. Hardness is sensitive to dislocation density due to work hardening but is practically not affected by vacancies while positron annihilation is sensitive both to dislocations and vacancies. Our investigations revealed that ultra fine grained copper contains dislocations and vacancy clusters created by agglomeration of deformation-induced vacancies. Average size of vacancy clusters increases with increasing radial distance from the centre of the sample due to higher production rate of vacancies resulting in larger clusters. During high pressure torsion deformation microhardness increases firstly at the periphery of the sample due to the highest imposed strain. With increasing number of high pressure torsion revolutions the hardness increases also in the centre and finally becomes practically uniform across the whole sample indicating the homogeneous distribution of dislocations. Doppler broadening mapping revealed a remarkable increase of $S$ parameter at the sample periphery due to larger size of vacancy clusters. The $S$ parameter remains significantly enhanced at the periphery even after 25 revolutions. Hence, contrary to dislocation density spatial distribution of vacancy clusters is far from being uniform even after prolonged high pressure torsion deformation.
1. Introduction

Severe plastic deformation (SPD) [1] enables to achieve extreme grain refinement down to nanoscopic dimensions leading to the formation of ultra fine grained (UFG) structure [2]. UFG materials with grain size below ~500 nm are characterized by highly non-equilibrium structure with high density of grain boundaries and high density of defects [1]. This leads to unique physical properties, in particular abnormally high diffusion activity [3] and very attractive mechanical properties characterized by high strength and sufficient ductility [4]. High-pressure torsion (HPT) [1,5] is the most efficient SPD technique for grain refinement. The principle of HPT technique is illustrated in Fig. 1a. The sample is placed between two anvils and compressed by a high pressure \( p \). One of the anvils is slowly rotating. Hence the sample is strained by torsion under a high pressure of several GPa. Bulk UFG materials with grain size typically around 100 nm can be produced by this technique [1,5]. Samples prepared by HPT are typically disk shaped with the thickness up to a few mm and the diameter up to 20 mm. In HPT deformation the strain increases with the radial distance \( r \) from the centre of the sample corresponding to the rotation axis, see Fig. 1b. The equivalent von Misses strain \( \varepsilon \) imposed by HPT deformation is given by [6]

\[
\varepsilon = \frac{2\pi rN}{h\sqrt{3}},
\]

where \( r \) is distance from the centre, \( h \) is the thickness of sample disk and \( N \) is the number of HPT revolutions. Because of non-uniform strain during HPT it is important to examine the homogeneity of UFG structure and the spatial distribution of deformation-induced defects across the sample disk.

\[\text{Figure 1.}\ (a)\ \text{Schematic illustration of HPT deformation; (b) depiction of HPT deformed specimen with diameter} \ r \ \text{and thickness} \ h; (c) \text{an image of HPT deformed Cu sample studied in this work.}\]

The aim of the present work is to characterize the homogeneity of microstructure and the spatial distribution of defects in HPT-deformed Cu. Positron annihilation spectroscopy (PAS) was employed for mapping of the spatial distribution of defects. This was performed using two PAS techniques: (i) positron lifetime (LT) spectroscopy and (ii) Doppler broadening (DB) of annihilation radiation. The LT spectroscopy enables to identify defects present in the sample and to determine their concentrations. But LT measurements with conventional positron source are time consuming and take usually at least one day. On the other hand measuring period of ~ 30 min is enough to accumulate a DB spectrum with statistics sufficient for precise determination of the line shape parameters \( S \) and \( W \). As was demonstrated in Ref. [7] the \( S \) parameter can be used for non-destructive mapping of the
spatial distribution of defects. In this work spatially resolved PAS was combined with mapping by microhardness testing and direct observation of microstructure by transmission electron microscopy (TEM).

2. Experimental

Cu (99.95% purity) specimens were subjected to room temperature quasi-constrained HPT straining using the compressive pressure of 4 GPa. The number of HPT revolutions was gradually increased from 1 to 25. HPT-deformed specimens were disk shaped with the diameter of \( \approx 9 \) mm and the thickness of \( \approx 0.3 \) mm, see Fig. 1c.

The homogeneity of microstructure was characterized by Vickers microhardness (HV) testing performed using STRUERS Duramin 300 hardness tester with load of 100 g applied for 10 s. HV measurements were performed on a perpendicular grid with incremental spacing of 0.5 mm. Colour-coded HV maps were constructed to provide pictorial displays of microstructure homogeneity across the sample disk. HPT-deformed Cu samples were polished to mirror like quality for HV investigations.

PAS investigations were performed using a \( ^{22}\text{NaCO}_3 \) positron source with activity of 1 MBq. The source spot was carefully deposited on a 2 \( \mu \)m thick mylar foil so that the spot diameter was smaller than 1 mm.

A digital spectrometer [8] with time resolution of 145 ps (FWHM of resolution function) was employed for LT spectroscopy. At least \( 10^7 \) positron annihilation events were collected in each LT spectrum which was decomposed into exponential components by a maximum likelihood code [9]. A well annealed (850 \(^\circ\)C) pure Cu reference sample characterized by a positron lifetime of 114 ps was used to determine the source contribution to the LT spectra. It consisted of two components with lifetimes 368 ps and 1.5 ns and corresponding relative intensities 8 and 1%. These contributions come from positrons annihilated in the \( ^{22}\text{NaCO}_3 \) source spot and the covering mylar foils, respectively, and were always subtracted from the spectra. Since it took one day to accumulate single LT spectrum with sufficient statistic it was not feasible to perform two-dimensional mapping of the samples by LT measurements. Instead we made ‘line scans’ when the positron source was moved along a line from the centre towards the edge and LT measurements were performed at various distances from \( r \) from the centre as illustrated in Fig. 2a.

\[ \text{(a)} \quad \text{(b)} \quad \text{(c)} \]

Figure 2. Schematic illustration of (a) line scan performed by LT spectroscopy; (b) DB/HV mapping performed across the whole sample; (c) regions cut from the sample for preparation of the foils for TEM, i.e. the centre region at \( r = 0 \) and the periphery region centred at \( r = 3 \) mm. Note that for better visibility Figs. 2(a) and 2(b) are not in scale, the actual mesh was denser.

DB of annihilation radiation was measured by a high purity germanium (HPGe) detector with energy resolution of 1.30 keV at 511 keV. At least \( 5 \times 10^5 \) counts were accumulated in the annihilation peak in each spectrum. DB of the annihilation peak was evaluated using the line shape \( S \) parameter.
The central region for calculation of the $S$ parameter was chosen symmetrically around the 511 keV annihilation peak from 510.07 to 511.93 keV. All $S$ values presented in this work were normalized to the $S$ parameter $S_0 = 0.5045(5)$ measured in the centre of the Cu sample subjected to one HPT revolution. As shown schematically in Fig. 2b the DB measurements were performed on a perpendicular grid with incremental spacing of 0.5 mm by placing the positron source spot to various nodes of the grid using a micrometer $x$-$y$ shift. The uncertainty in the position of centre of the positron source was approximately $\sim 0.1$ mm. It took around 30 min to measure each point. Colour coded maps of $S$ parameter values were constructed in the similar way as in the case of HV mapping. PAS investigations were performed directly on HPT-deformed samples without any surface treatment. In a single HPT-deformed sample selected from the series a sub-surface layer with thickness of $\approx 30 \, \mu$m was removed by chemical etching in order to check whether the density of deformation-induced defects changes with depth. The PAS results for the etched sample were virtually the same as those for the sample prior to etching. This testifies that there is no significant change of defect density with the depth from the surface.

TEM observations were carried out on a JEOL JEM 2000 FX electron microscope operating at 200 kV. Foils for TEM were prepared by electrolytic polishing in STRUERS TENUPOL 5 jet-polishing unit using 50% H$_3$PO$_4$ at 10 °C. As shown schematically in Fig. 2c foils for TEM investigations were cut (i) from the centre of the sample disk ($r = 0$) and (ii) from region at the periphery (the centre of the foil was at $r = 3$ mm) in order to compare the microstructure in these two regions.

3. *Ab-initio* theoretical calculations

Density functional theory was employed for *ab-initio* calculations of lifetimes of positrons trapped at various point defects in Cu. Positron density was calculated within the so-called standard scheme employing the atomic superposition (ATSUP) method [10]. The electron–positron correlation was treated using the local density approximation (LDA) according to the parameterization by Boroniński and Nieminen [11]. The calculations were performed in 256 Cu atom based supercells. Point defects (vacancies and vacancy clusters) were modelled simply by removing the corresponding number of atoms from the supercell.

![Figure 3](image)

**Figure 3.** (a) Development of positron lifetimes with increasing number of HPT revolutions; (b) dependence of the intensity $I_2$ of positrons trapped at vacancy clusters on the number of HPT revolutions. The sample was measured in the centre ($r = 0$) and at the periphery ($r = 3$ mm).

4. Results and Discussion

HPT deformed Cu samples exhibit two component LT spectra. Lifetimes of both these components are significantly higher than the bulk Cu lifetime $\tau_B = 114$ ps [12] indicating that both these
components come from positrons trapped at defects. No free positron component could be resolved in the LT spectra. This testifies to the very high density of defects even after the first HPT revolution leading to saturated positron trapping.

The shorter component with lifetime $\tau_1 \approx 165$ ps represents a contribution of positrons trapped at dislocations \[12,13\]. Thus, dislocations created during SPD represent the dominating type of defects in HPT-deformed Cu samples. The longer component with lifetime $\tau_2$ falling in the range 240 – 320 ps can be attributed to positrons trapped at vacancy clusters created by agglomeration of vacancies \[13,14\]. Plastic deformation introduces vacancies by non-conservative motion of dislocations. The most common mechanism of vacancy creation during plastic deformation is climbing of edge dislocations which results in the formation of chains of vacancies. Since vacancies in Cu are mobile at room temperature \[15\] they either disappear by diffusion to sinks at grain boundaries and surface or agglomerate into small vacancy clusters which are stable at room temperature.

The development of dislocations and vacancy clusters during HPT processing is presented in Fig 3. Lifetimes $\tau_1$ and $\tau_2$ resolved in LT spectra are plotted in Fig. 3a as a function of the number $N$ of HPT revolutions. The lifetime of the dislocation component remains constant during HPT processing except of statistical scattering while the lifetime $\tau_2$ of the cluster contribution slightly increases with increasing number of HPT revolutions. The relative intensity $I_2$ of the cluster contribution plotted in Fig. 3b increases with increasing number of HPT rotations as well. The samples were measured in the centre ($r = 0$) and at the periphery ($r = 3$ mm). Both the lifetime $\tau_2$ and the intensity $I_2$ of the cluster component are remarkably higher at the periphery than in the centre. This testifies that vacancy clusters at the periphery are larger than in the centre.

In order to determine the size of vacancy clusters experimental data were compared with \textit{ab-initio} theoretical calculations. Fig. 4a shows the calculated lifetime of positrons trapped at vacancy clusters of various sizes. The size of vacancy clusters is expressed as number of vacancies $N_V$ in the cluster. The lifetime of trapped positrons obviously increases with increasing cluster size. For small vacancy clusters positron lifetime steeply increases while for large clusters in gradually saturates. By comparing the experimental lifetime $\tau_2$ with the theoretical calculations in Fig. 4a the mean size of vacancy clusters in HPT Cu samples was determined.

Fig. 4b shows the dependence of the mean size of vacancy clusters on the number of HPT turns. Vacancy clusters at the centre consist of 4–5 vacancies, i.e. they are smaller than the clusters at the

\[\text{Figure 4.} \ (a) \text{ Calculated positron lifetime for vacancy clusters of various sizes plotted as a function of the cluster size (expressed as the number of vacancies } N_V \text{ in the cluster);} \ (b) \text{ dependence of the mean size of vacancy clusters on the number of HPT revolutions. The sample was measured in the centre } (r = 0) \text{ and at the periphery } (r = 3 \text{ mm).}\]
periphery, which consist of 8–9 vacancies. No significant changes in the size of vacancy clusters with increasing number of HPT turns were observed.

The vacancy production rate \( \Pi \) at low temperature plastic deformation is governed by the strain rate \( \dot{\varepsilon} \) [16]

\[
\Pi = \alpha \frac{\sigma \Omega_0}{E_f} \dot{\varepsilon},
\]

where the coefficient \( \alpha \approx 0.1 \) [17], \( \sigma \) is the applied stress, \( \Omega_0 \) the atomic volume and \( E_f \) the vacancy formation energy. Since in torsion deformation the strain rate increases with the radial distance from the centre of the sample the production rate of vacancies at the periphery is higher than in the centre. This leads to the formation of bigger vacancy clusters at the periphery.

Because of saturated positron trapping the ratio \( I_2/I_1 \) of the relative intensities of positrons trapped in vacancy clusters and at dislocations is directly proportional to the ratio of defect concentrations:

\[
\frac{I_2}{I_1} = \frac{v_{VC}c_{VC}}{v_D\rho_D},
\]

where \( c_{VC} \) denotes the concentration of vacancy clusters and \( \rho_D \) stands for the dislocation density. The symbols \( v_{VC} \) and \( v_D \) denote the specific positron trapping rate to vacancy clusters and dislocations, respectively. The specific positron trapping rate for dislocations in Cu \( v_D = 0.6 \times 10^4 \text{ m}^2 \text{ s}^{-1} \) was estimated in Ref. [18]. The specific positron trapping rate for a small vacancy cluster consisting of \( N_V \) vacancies \( (N_V \leq 10) \) is proportional to the number of vacancies in the cluster. Hence, Eq. (3) can be rewritten as

\[
\frac{I_2}{I_1} = \frac{N_V v_V c_{VC}}{v_D\rho_D},
\]

where \( v_V = (1.2 \pm 0.2) \times 10^{14} \text{ at. s}^{-1} \) [19] is the specific positron trapping rate for a monovacancy in Cu. The dislocation density \( \rho_D \) in HPT-deformed Cu samples was determined in Ref. [13] by X-ray line profile analysis and was found to increase with increasing number of HPT revolutions. Using the \( \rho_D \) values determined in Ref. [13] the concentration of vacancy clusters was calculated from Eq. (4) and is plotted as a function of the equivalent strain \( \varepsilon \) in Fig. 5a. One can see in the figure that \( c_{VC} \) increases at low strains \( (\varepsilon \leq 10) \), then it exhibits a plateau followed by further increase at very high strains \( (\varepsilon \geq 200) \). Increasing number of deformation-induced vacancies leads either to increase of the average size

![Figure 5](image-url)
of vacancy clusters and/or to increase of their concentration. However, only the latter effect is visible in Fig. 5a. Hence, in the ‘plateau region’ (10 < \( \varepsilon \) < 200) the increasing concentration of deformation-induced vacancies most probably leads to the formation of bigger vacancy clusters without significant change of their concentration. Since the vacancy clusters consist on average of \( N_V \) vacancies the net concentration of deformation-induced vacancies which agglomerated into clusters was estimated as \( c_V \approx N_V c_{VC} \) and is plotted in Fig. 5b as a function of the equivalent strain \( \varepsilon \). One can see in the figure that \( c_V \) continuously increases with increasing strain testifying that plastic deformation applied during HPT processing introduces continuously vacancies into the sample.

Hence, there are two factors influencing vacancy clusters in HPT-deformed Cu: (i) the strain rate and (ii) the total equivalent strain. Higher strain rate leads to formation of bigger vacancy clusters due to higher production rate of vacancies. Higher equivalent strain leads to higher concentration of deformation-induced vacancies and consequently to formation of vacancy clusters which are larger in size or are present in higher concentration.

\[ \text{Figure 6. Results of a ‘line scan’ performed by LT spectroscopy across the radius of UFG Cu deformed by to 2 HPT revolutions: dependence of (a) lifetimes resolved in LT spectra and (b) the relative intensity } I_2 \text{ of positrons trapped at vacancy clusters on the radial distance } r \text{ from the centre of the sample disk. Solid lines are intended to guide the eye only.} \]

Detailed mapping of spatial distribution of defects across the radius of sample was performed by LT spectroscopy for the UFG Cu sample deformed by 2 HPT turns. Fig. 6a shows lifetimes resolved in LT spectra plotted as a function of the radial distance from the centre. Saturated trapping at dislocations and in vacancy clusters was observed in all points measured. The dependence of the relative intensity \( I_2 \) of positrons trapped at vacancy clusters on the radial distance from the centre is shown in Fig. 6b. Obviously both the lifetime \( \tau_2 \) and the intensity \( I_2 \) of the cluster contribution gradually increase with increasing distance from the centre. Hence, the average size of vacancy clusters gradually increases from the centre of the sample towards the edge due to increasing production rate of vacancies. Since larger vacancy clusters are more efficient positron traps characterized by higher specific positron trapping rate, the fraction of positrons trapped at these clusters increases with \( r \) even if the concentration of vacancy clusters remains unchanged.

Fig. 7 shows results of HV mapping of UFG Cu samples subjected to various numbers \( N \) of HPT revolutions. The HV maps exhibit axial symmetry with respect to the centre of the sample as expected for deformation by torsion. One can see in the figure that HV at the periphery quickly increases and becomes saturated at the value \( HV \sim 155 \). On the other hand, HV in central region of the sample is significantly lower. With increasing number of HPT revolutions the region with enhanced hardness...
gradually extends from the periphery towards the centre of the sample disk. Finally in the sample deformed by 25 HPT revolutions HV becomes almost uniform practically across the whole sample except for a small region in the centre of the sample.

The average HV at various distances from the centre of the sample were calculated by angular averaging the values measured at the same distances from the centre. Fig. 8a shows the dependence of HV on the equivalent strain $\varepsilon$. The HV values for samples subjected to various numbers of HPT turns fall on a common sigmoid ‘master curve’ when plotted versus the equivalent strain. Small scattering between various samples is likely caused by uncertainties of centring the sample disk during HPT processing. One can see in the figure that HV increases with increasing strain and gradually saturates at HV ~ 155 for $\varepsilon \geq 200$.

Microstructure of samples deformed by various numbers of HPT revolutions was examined by TEM in order to correlate microstructure evolution with hardness variations. Each sample was probed

**Figure 7.** Colour coded map constructed from HV values measured on a rectangular grid across the sample disk for UFG Cu deformed by various numbers $N$ of HPT revolutions.

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Microstructure of samples deformed by various numbers of HPT revolutions was examined by TEM in order to correlate microstructure evolution with hardness variations. Each sample was probed
in the centre and at the periphery as shown in Fig. 2c. TEM micrographs for the sample deformed by single HPT revolution are presented in Fig. 9. Heavily deformed structure with a high density of dislocations and a strong stress was observed both in the centre and at the periphery. Microstructure in the centre exhibits features typical for initial stages of dislocation re-arrangement, i.e. the formation of cells with low dislocation density separated by dislocation walls containing significantly higher dislocation density. On the other hand, more refined microstructure was observed at the specimen periphery. Dislocation walls become sharper and transform into sub-grain boundaries. Several grains with dislocation-free grain interiors separated by sharp grain boundaries can be observed in Fig. 9b. Hence, TEM investigations revealed significantly different microstructure in the centre and at the periphery. Stronger grain refinement at the periphery is in agreement with higher strain and explains also enhanced hardness at the periphery.

![Figure 8](image-url)

**Figure 8.** The dependence of HV (a) and the $S$ parameter (b) on the equivalent strain $\varepsilon$. Solid lines are guides for the eye.

The microstructure of the sample subjected to 3 HPT revolutions is shown in Fig. 10. The centre of the sample (Fig. 10a) is characterized by dislocation cells and partially developed sub-grains, i.e. the microstructure similar to that observed in the centre after one HPT turn, cf. Fig. 9a. Two types of grains can be recognized at the periphery of the sample (Fig. 10b): (i) almost dislocation-free grains with sharp equilibrium grain boundaries and (ii) grains separated by non-equilibrium grain boundaries with diffuse contrast. The mean grain size at the periphery was estimated as $\sim$ 500 nm.

Fig. 11a shows the microstructure in the centre of sample subjected to 15 HPT turns. The microstructure in the centre consists of dislocation cells and sub-grains separated by tangled dislocations. The periphery of the sample shown in Fig. 11b exhibits refined structure with the mean grain size of 200-300 nm. Similarly to the sample subjected to 3 HPT revolutions one can distinguish in this specimen grains without dislocations with sharp grain boundaries and grains containing dislocations separated by non-equilibrium grain boundaries.

Hence, from TEM investigation it becomes clear that microstructure in the centre of sample differs significantly from that at the periphery even after 15 HPT revolutions. X-ray line profile analysis revealed that dislocation density exhibits only moderate variations across the HPT samples [13,20]. Hence, the enhanced HV at the periphery is most probably caused by finer microstructure with higher density of grain boundaries (so called grain boundary strengthening). It has to be noted that hardness is related to the mean grain size $d$ by the Hall-Petch relation [21,22] $HV \sim d^{1.2}$.

Colour coded maps constructed from the $S$ parameters measured by DB spectroscopy on the nodes of a rectangular mesh across the sample disks deformed by various number of HPT turns are displayed in Fig. 12. Similarly to HV mapping also the $S$ parameter maps exhibit axial symmetry with respect to the centre of the sample. The $S$ parameter map for the sample deformed by a single HPT turn is plotted...
in Fig. 12a. The $S$ parameter values are clearly enhanced at the periphery of the sample disk. In the sample deformed by 3 HPT revolutions the $S$ parameter at the periphery increases even more and saturates at $S/S_0 \sim 1.18$, see Fig. 12b. Further increase in the number of HPT revolutions leads to increase of $S$ also in the central region, but a pronounced difference between the centre and the periphery remains even in the sample deformed by 25 HPT turns, see Fig. 12d.

![Figure 9](image)

**Figure 9.** Bright field TEM micrographs of Cu subjected by single HPT revolution: (a) centre of the sample ($r = 0$); (b) periphery ($r = 3$ mm).

The HV and $S$ parameter mapping should be considered as mutually complementary techniques providing different point of views on the UFG microstructure. HV is influenced mainly by the grain size (grain boundary strengthening) and the dislocation density (work hardening) while DB spectroscopy is sensitive to the spatial distribution of dislocations and also to vacancy clusters which have relatively low impact on hardness. The general trend in HV and $S$ parameter is similar – i.e. both increase with increasing radial distance from the centre due to enhanced defect density and decreasing grain size. But different sensitivity of HV and DB technique leads to remarkable differences between HV and $S$ parameter maps which can be clearly seen by comparing Figs. 7 and 12. The sample subjected to 25 HPT revolutions is characterized by almost uniform HV, see Fig. 7f, while the $S$ parameter in this sample remains significantly higher at the periphery compared to the centre, see Fig. 12d. This difference is mainly due to vacancy clusters which are substantially bigger at the periphery than in the centre due to higher production rate of vacancies. Hence, although HV mapping showed only a slight difference between the centre and the periphery in the sample subjected to 25 HPT revolutions, DB mapping revealed that lateral distribution of vacancy clusters is far from being uniform and clusters formed at the periphery are always bigger than in the centre.
Figure 10. Bright field TEM micrographs of Cu deformed by $N = 3$ HPT revolutions: (a) centre of the sample ($r = 0$); (b) periphery ($r = 3$ mm).

The $S$ parameters obtained for each sample by angular averaging of the values measured at the same distance from the centre are plotted as a function of the equivalent strain in Fig. 8b. By comparing of Figs. 8a and 8b it is clear that there is a striking difference between the development of HV and $S$ parameter values with strain. While HV values for all samples subjected to various number of HPT revolutions fell on a common ‘master curve’ the $S$ parameter behaves in a completely different way. For each sample $S$ strongly increases at the periphery. This increase is less pronounced in the sample subjected to a single HPT revolution. However, in samples deformed by $N \geq 3$ HPT turns the $S$ parameter behaviour is almost the same independently of strain. Hence, except of a single common ‘master curve’, the $S$ parameter values when plotted versus the total strain show separate curves for each sample. This indicates that variations of the $S$ parameter across the HPT-deformed samples are predominantly influenced by changes in the size of vacancy clusters. As it was explained in the previous discussion the size of vacancy clusters is determined mainly by the strain rate, which depends on the distance from the centre of the sample irrespective of the total strain. Thus, $S$ parameter maps reflect predominantly variations of the size and the concentration of vacancy clusters while HV maps reflect mainly variations in the grain size.
Figure 11. Bright field TEM micrographs of Cu deformed by $N = 15$ HPT revolutions: (a) centre of the sample ($r = 0$); (b) periphery ($r = 3$ mm).

Figure 12. Colour coded maps of $S$ parameters constructed from values measured on a rectangular grid across the sample disk for UFG Cu deformed by various numbers $N$ of HPT revolutions.
5. Conclusions
Two types of lattice defects were found in ultra fine grained Cu prepared by high pressure torsion: (i) dislocations and (ii) small vacancy clusters formed by agglomeration of deformation-induced vacancies. The size of vacancy clusters increases from centre of the sample towards its periphery due to increasing strain rate leading to higher production rate of vacancies. Homogeneity of ultra fine grained structure and spatial distribution of defects were mapped by spatially resolved microhardness testing and positron annihilation spectroscopy. Both these techniques are complementary and reflect different aspects of the microstructure. Hardness mapping visualizes predominantly variations of the grain size while mapping by the $S$ parameters shows mainly changes in the size and the concentration of vacancy clusters. The specimen deformed by 25 revolutions exhibits practically uniform hardness across the whole sample disk while the $S$ parameter is enhanced at the periphery and lowered in the central region. This testifies that the mean grain size became comparable in the whole sample after sufficient number of revolutions but vacancy clusters are always bigger at the periphery than in the centre.

Acknowledgements
This work was supported by the Czech Science Foundation (project P108-13-09436S). One of the authors (M.J.) acknowledges financial support by the same Foundation under the project 14-36566G.

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