suitable properties: common and stable phase in wide *p–T* field, good optical and electrical properties, etc. In several cases beside the constituting elements, Fe and S, unusual elements, like Au, Ag, Si, Pb, O could be detected by spectroscopies, whose structural interpretation has not been revealed yet.

We studied a pyrite sample from Sudbury Igneous Complex that has an unusual octahedral morphology and contains significant (several at%) amount of Si. Our aim is to interpret the Si in the crystal structure applying high resolution transmission electron microscopy (HRTEM), energy-filtered TEM, EDX spectrometry. For accurate scattered intensity retrieval, we applied precession electron diffraction (PED) technique (Vincent & Midgley, 1994) using Digistar system (NanoMegas). Since the multiple/dynamical scattering is very intensive even in very thin (<100 nm) pyrite sample according to multislice simulations (JEMS software, Stadelman, 1987), we applied relatively high, 2.55° precession angle for intensity acquisition. The reciprocal cell was reconstructed from tilt series of diffraction patterns. HRTEM images were acquired mainly in [100] and [110] projections and were corrected for the contrast transfer function (CTF) and as well as by the measured PED intensities (Fig. 1) Pyrite is known to have a cubic cell with $a_0 \sim 5.4$ Å and *Pa*-3 space group (Oftedal, 1928). According to *Pa*-3 I_{010}, and I_{030} should be 0. However, they usually appear on the SAED patterns of [001] and $[10\bar{1}]$ projections because of the strong dynamical effects. On PED patterns of Si-containing pyrite we observed both the 010 and the 030 reflections. We found that in [001] projections the two octahedral layers parallel to (010) and connected to each other by a glide plane are not equal, as it can be seen on projected charge density map acquired from corrected HRTEM images (Fig. 1).

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FLUCTUATION ELECTRON MICROSCOPY OF METALLIC GLASSES BY TILTED DARK-FIELD IMAGING

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Keywords: fluctuation electron microscopy, bulk metallic glasses, medium range order

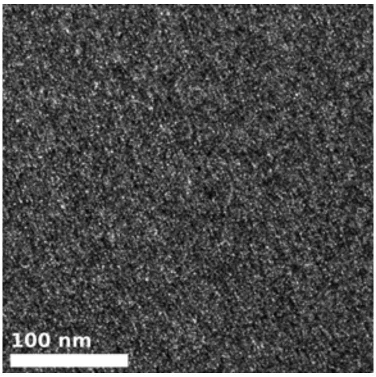

Fig. 1. TEM dark-field image of CuZrAlAg bulk metallic glass showing the characteristic speckle pattern which gives rise to the signal in the normalized variance curve. Their size corresponds to the results of the variable resolution FEM (cf. Fig. 4)

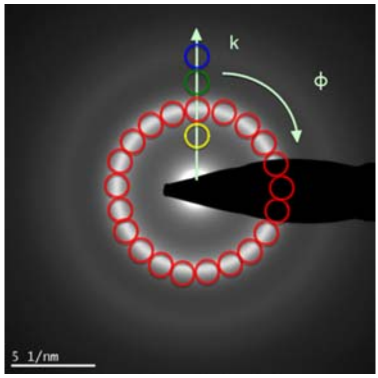

Fig. 2. Schematic illustration of dark-field image acquisition by sampling the reciprocal space. The objective aperture (indicated by the circles) samples the *k* space by tilting and rotating of the incident beam

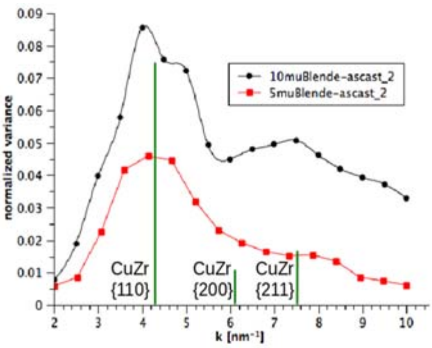

Fig. 3. The normalized variance curve as obtained from the CuZrAlAg bulk metallic glass shows peaks at *k* values similar to those corresponding to B2 CuZr

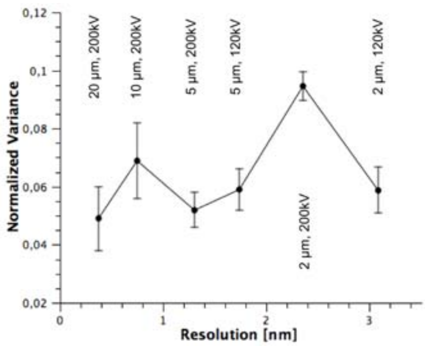

Fig. 4. Variable resolution FEM using the peak maxima of the normalized variance reveales medium range order length scales of about 0.7 and 2.3 nm

Bulk metallic glasses (BMG) show attractive properties as very high strength and high elastic limit, which are closely related to their atomic arrangement. Therefore, a quantitative description of the structure of BMG is necessary to tailor their properties. Unlike crystalline materials, the structure of BMG is characterized by the lack of long-range periodicity as in an amorphous structure; still it is assumed that short-range order (SRO) as well as medium-range order (MRO) are present in BMG. Fluctuation electron microscopy (FEM) [1] is a diffraction technique to study atomic correlations of a BMG on the MRO scale.

Samples of CuZrAlAg, a BMG produced by copper mould suction casting, were prepared by electropolishing to obtain TEM foils. FEM experiments by taking tilted dark-field images were carried out in a TEM operating at 120 and 200 kV. The dark-field images show intensity variations due to local structural correlations on the MRO scale (cf. Fig. 1). In order to measure MRO, dark-field images taken at different scattering vectors *k* were analyzed statistically by calculating the normalized variance $V(k)$ of the image intensity $I(k, r)$ [1]: $V(k) = (\langle I(k, r)^2 \rangle / \langle I(k, r) \rangle^2) - 1$, where $\langle\,\rangle$ means averaging over sample position *r*.

In order to increase the reliability of $V(k)$ a set of dark-field images was taken by varying the scattering vector k and the angle Φ (cf. Fig. 2). The acquisition of dark-field images probing the reciprocal space between given values of k and Φ was automated by a script running under “Digital Micrograph”. A peak in the curve indicates that intensity speckles in the images measured by the normalized variance are especially pronounced at a given k value (4.2 nm^{-1}). The scattering vector of the maximum contains information of the MRO structure. It is interesting to note that in crystalline B2 ordered CuZr the strongest reflection corresponding to (110) has a very similar k value indicating some similarity in the atomic correlations as in the MRO of the BMG (cf. Fig. 3).

In order to get information on the correlation length sets of dark-field images with different spatial resolution (by changing objective aperture size and/or acceleration voltage) are recorded. Their corresponding calculated $V(k)$ curves show the same characteristics with a peak at 4.2 nm^{-1}; as an example the curves obtained from images taken with 5 and 10 µm objective apertures are shown in Fig. 3. Plotting the peak values of $V(k = 4.2\ nm^{-1})$ as a function of resolution yields a curve containing two maxima (cf. Fig. 4). Two maxima in V as a function of resolution indicate the presence of two MRO correlation lengths of about 0.7 and 2.3 nm in the amorphous structure. The observed MRO correlation lengths can be linked to the size of individual clusters and to the correlation between similarly oriented clusters.

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LOW-VOLTAGE TEM FROM 10 kV TO 25 kV

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Keywords: low-voltage TEM, biological samples

The application studies of new low-voltage transmission microscope LVEM25, working at 10–25 kV are presented. The LVEM25 was designed on the basis of experience with the low-voltage transmission electron microscopy at 5 kV [1, 2], which is intended for the study of samples with low contrast (organic matters). Many aspects were optimized during design of the instruments:

1. Maintaining relatively low voltage to keep up high contrast.
2. The use of such energy, which would open the possibility to increase the resolution of the system to the area of atomic (molecular) resolution using the monochromatization of the primary beam and Cs correction in future.
3. Practical standpoints – reasonable dimensions, resistance to external influences.