A METALLOGRAPHIC STUDY OF THE METEORITES: CAPABILITIES OF EBSD METHOD.

V. I. Grokhovsky, K. A. Badekha, E. V. Brusnitsyna, R. F. Muftakhetdinova, G. A. Yakovlev. Institute of Physics and Technology, Ural Federal University, Ekaterinburg, 620002, Russian Federation. E-mail: grokh47@mail.ru.

Introduction: A metallographic study of the meteorites structure is useful method of analysis for classification and the cooling rates estimation of an extraterrestrial metal both in the $\alpha \rightarrow \gamma$ transformation temperature range and at the spinodal decomposition temperature. Often, new analytical capabilities are tested using meteoritic metal. Researchers began to use many new techniques over the last quarter of a century: FIB, computer tomography, nanoindenting, EBSD, image analysis. EBSD method provides wide possibilities in acquiring local crystallographic information. Metallographic studies of extraterrestrial metal at the Ural Federal University were carried out since 1971. First works described metallic phases in lunar soil. Besides morphology and local chemical composition, one should have diffraction data for good phase identification. Registration of Kossel lines (X-ray diffraction) and Kikuchi lines can provide such information. It was Kossel technique that UrFU's researchers applied for determination of kamacite lattice parameters in particles of lunar soil from Luna 16 and Luna 20. The first demonstrations of EBSD on meteorites were given in works [1,2]. Here, examples of phase and orientation EBSD analysis for the identification of phases and structural transformations in a meteorite metal are presented.

Experimental: Samples were polished using standard metallographic polishing procedure followed by polishing using 0,04μm SiO_2 for 2 hr. EBSD studies were accomplished using FE-SEM SIGMA VP and SEM JEOL JSM-6490LV with EDS and EBSD units. Additionally, program CaRIne Crystallography 3.1 for obtaining stereographic projec-tions and modeling the crystals structure has been used.

Meteorites of various types were investigated: Chelyabinsk (LL5), Sikhote-Alin (IIAB), Chinga (Iron-ung), Gebel Kamil (Iron-ung), Hoba (IVB), Iquique (IVB), Cape of Good Hope (IVB), Bilibino (IIAB), Aliskerovo (IIIAB).

The EBSD method has a wide range of applications for solving problems in the study of various minerals of meteoritic matter. For example, roaldite $(Fe, Ni)_4N$ revealing is too difficult due to similarity of its morphology with rhabdite and Neiman bands, especially in the range size less than 1 μm. However, EBSD allowed to identify thin plates of roaldite in the Sikhote-Alin meteorite. The phase contrast map demonstrates the presence of kamacite, rabdite and roaldite. The character of roaldite morphology in Sikhote-Alin meteorite indicated that roaldite was formed after complete precipitation of rhabdite [3].

Haxonite was found in taenite area of the metal grain near taenite/kamacite boundary in Chelybinsk LL5 meteorite. EBSD analysis confirmed that light particles have taenite lattice and dark matrix have haxonite lattice (fig.1). Earlier, the cubic carbide in iron meteorites has been well described [4]. Previously [5], it was suggested, that graphite and carbides precipitated after finish of crystallization. Carbides formed at low temperature after kamacite and schreibersite. In our section graphite was not found.

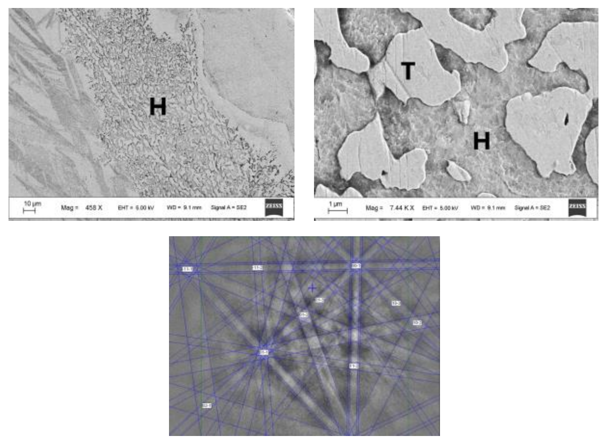

Fig. 1. Haxonite (H) assosiasted with taenite (T) in Chelyabinsk LL5 meteorite.

Also, this technique allowed to suggest the origin of the Schlieren bands in ataxites. Schlieren effect in the ataxites was known for a long time, but the origin of this bands was not clear. Each investigated ataxite demonstrate a set of three main bcc orientations which retained in the neighboring Schlieren bands while the dominant bcc orientation in these bands was different. The dominant bcc orientations in the Schlieren bands coincided with the orientation of kamacite spindles. Therefore, the Schlieren bands were drawn out along the Widmanshtätten direction. The presence of retained γ phase after martensite transformation (γ_R) as well as exsolved γ phase from martensite (γ_E) was shown earlier [6, 7]. We observed that orientation of γ_R phase was the same in dark and light bonds. This fact excludes twinning origin of the bands. It was further shown that planes (111) for γ_R and (011) for α were parallel. The directions [1$\bar{1}$0] for γ_R and [100] for α in both dark and light bands were also parallel that was close to Nashiyama-Vasserman martensite orientation relationship. These results demonstrated that taenite decomposition in Chinga ataxite was by martensite type reaction: $\gamma_R \rightarrow \alpha_2 + \gamma_R \rightarrow \alpha' + \gamma_E + \gamma_R$. Thus, we can conclude that Schlieren bands appeared due to formation of different crystallographic set of submicroscopic products during martensite transformation [8, 9].

EBSD analysis serves as an excellent tool for solving problems of changing orientation without changing chemical and phase composition. Thus, in the shock-induced samples of the Sikhote-Alin meteorite, the regions of contact melting at the kamacite–rhabdite boundary were found around some rhabdites. Concentration of Ni was not changed in comparison with the initial kamacite matrix. EBSD analysis demonstrated misorientation of the formed rim around the rhabdite and the rhabdite itself. The phase map indicates the fcc lattice (fig.2, 3). The eutectic liquid in these regions formed during a local heating. EDS analysis of these regions revealed a decrease of phosphorus content in comparison with that in rhabdite. The phase and orientation maps demonstrated polycrystalline α-Fe(Ni). The area of contact melting after heating (above the melting point of 950°C) and rapid cooling transformed to the supersaturated solid solution of P in the kamacite (α-Fe(Ni)+P) [10].

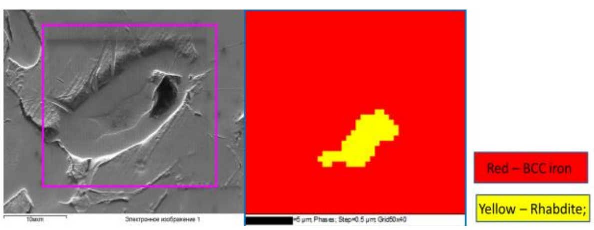

Fig.2. SEM image of contact melting zone and EBSD phase mapping.

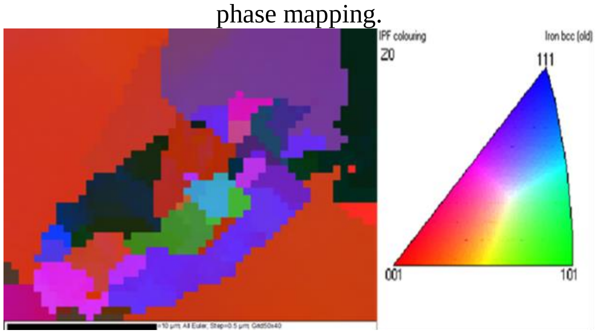

Fig.3. Orientation map of contact melting zone according to EBSD data.

EBSD analysis is almost the only method for proving the passage of α→ε transformation. We have studied microstructural deformation-induced changes and phase transformations in the material of the Sikhote-Alin iron meteorite (IIAB) after loading by spherically converging shock waves. The results obtained by the method of electron backscatter diffraction, as well as the data of local chemical analysis unambiguously indicate the presence of regions experiencing polymorphic α→ε and ε→α transitions in the loaded sample [11].

Structural changes in kamacite were investigated in iron meteorites of ancient fall (Aliskerovo IIIAB, Bilibino IIAB) with EDS and EBSD units. Samples which were significantly affected by climate were chosen for research of climatic factors. All of them demonstrate uncompleted recrystallization. It was noticed that recrystallization started from the kamacite-rhabdite boundaries in the Bilibino meteorite and from the kamacite-schreibersite boundaries in the Aliskerovo meteorite. There are strongly etched sites in the recrystallized zones. One can suggest that these sites are traces of former boundaries. It is possible to think that the boundaries were moving with jumps because of the position of these sites in the recrystallized zone. Also, it was noticed that there is a net of cracks before the recrystallization reaction front. A possible reason for this phenomenon is a wedge of extra material which generates an elastic stress field in the vicinity of the grain boundary [12]. All these phenomena can be explained using the grain boundary Kirkendall effect: the boundary shift is the result of the different concentrations of vacancies between the boundary sides [13].

Acknowledgement: This work was supported in part by the Ministry of Education and Science of the Russian Federation (The projects 5.4825.2017/6.7, 5.3451.2017/4.6) and the Act 211 of the Government of the Russian Federation. Contribution to the study from G.A.Y. was funded by the RFBR according to the research Project No. 16-38-00532 mol_a.

References: [1] Zucolotto M.E. and Pinto A.L. (2000) *Meteoritics & Planet. Sci., 35*, A180. [2] Zucolotto M.E. and Pinto A.L. (2001) *Meteoritics & Planet. Sci., 36*, A234. [3] Uymina K.A. et al. (2010) *Meteoritics & Planet. Sci., 45*, A205. [4] Scott E. R. D. (1971) *Nature Physical science, 229*, 6–62. [5] Scott E. R. D. (2012) *LPS XXXXIII*, Abstract #2671. [6] Nolze G. and Geist V. (2004) *Cryst. Res. Technol. 39*, 343–352. [7] Goldstein J. I. and Michael J. R. (2006) *Meteoritics & Planet. Sci., 41*, 553–570. [8] Grokhovsky V. I. et al. (2008) *Meteoritics & Planet. Sci., 43*, A50. [9] Badekha K. A. (2012) *Meteoritics & Planet. Sci., 47*, A49. [10] Gizzatullina R. F. et al. (2014) *Meteoritics & Planet. Sci., 49*, A137. [11] Muftakhetdinova R.F. et al. (2016) *Technical Physics, 61(12)* 1830–1834. [12] Klinger L., Rabkin E. (2011) *Acta Materialia, 59*, 1389–1399. [13] Yakovlev G. A (2013) *Meteoritics & Planet. Sci., 48*, A382.