Figure 2. Diversity of mineral species from the series of lillianite homologues with N = 4: Star - staročeskéite, Gus - gustavite, UN - undersubstituted gustavite, Ter - terrywallaceite, Fiz – fizelyite, Ram – ramdohrite, And IV – andorite IV, Nak – nakaseite (Cu-rich andorite VI).

2), it is necessary to determine the ranges of substitution (substitution limits) within which the mineral is defined. Empirically it was determined that the name staročeskéite would be valid for a lillianite structure with composition $Ag_xPb_{3-2x}Bi_ySb_{2+x-y}S_6$ with the boundaries $\frac{1}{2}$ x 0.8, and $1-\frac{1}{2}x$ y 2, where the parameter x = Ag content = L% and y = total Bi content. Thus we concluded that for staročeskéite to exist, there must between 20 to 50 % occupation of $M2$ site by Pb, apart from fully occupied $M3$ site. Other Pb concentrations in $M2$ site lead to different minerals.

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2. R. Pažout, J. Sejkora J (2018) Staročeskéite, $Ag_{0.70}Pb_{1.60}(Bi_{1.35}Sb_{1.35})_{2.70}S_6$, from Kutná Hora, Czech Republic, a new member of lillianite homologous series. *Mineralogical Magazine*, 1-26. doi:10.1180/minmag.2017.

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L5

COMPARISON OF RESIDUAL STRESSES DETERMINED USING DIFFERENT METHODS

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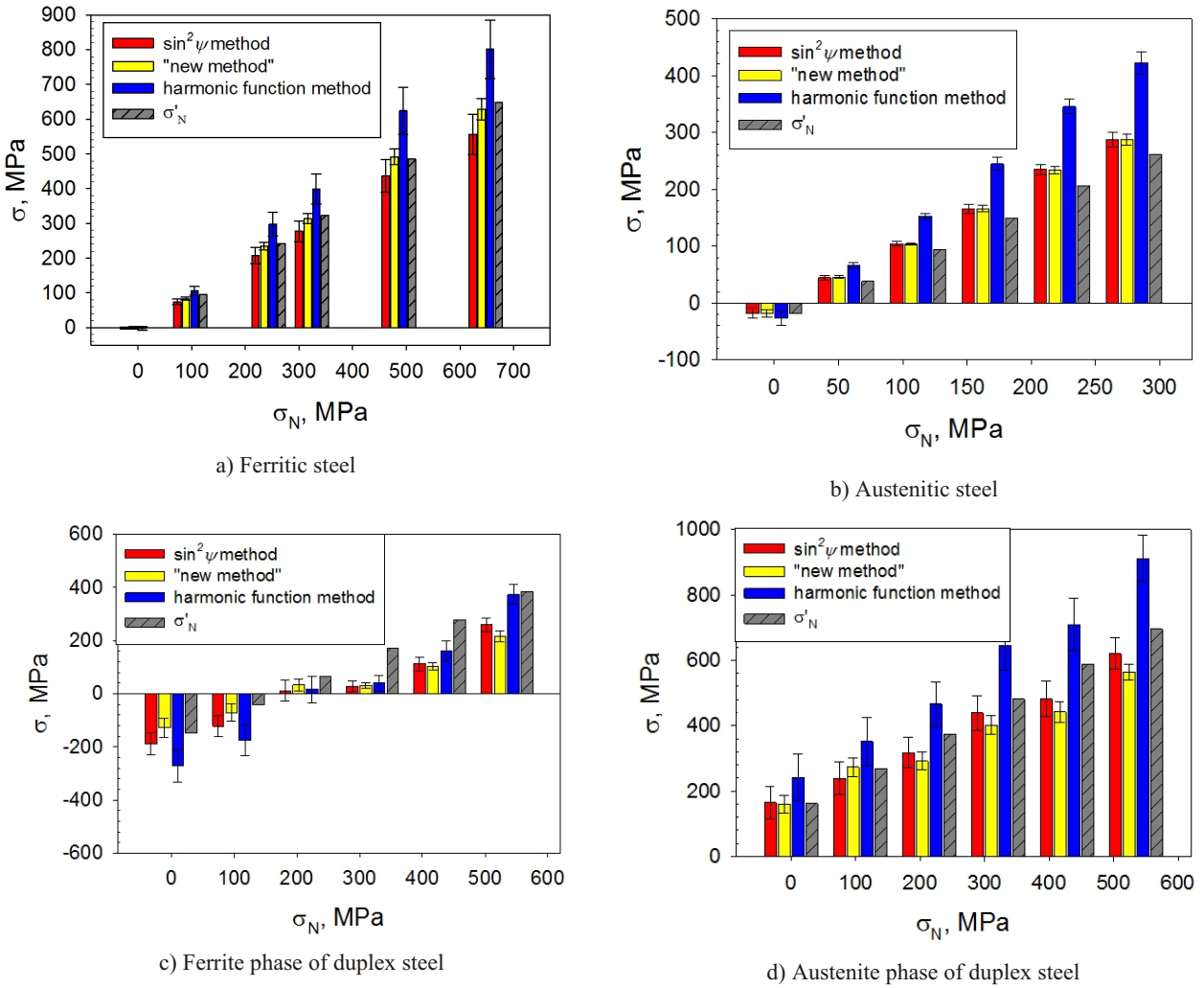
The majority of hitherto practically used diffraction measurements methods and algorithms for residual stresses calculation assume case of isotropic (non-textured) poly-cr ystalline material. Due to the comparatively frequent existence of preferred orientation (texture), not only in metals, it is more than desirable to have at disposal a method, procedure and even a computation algorithm for proper and correct residual stresses determination. For this purpose, a new method was used for determination of residual stresses without neglecting the texture.

For determination of residual stresses, three methods were used: standard $\sin^2$ method, method of harmonic function [1] and new method based on a model by Dölle [2, 3]. Contrary to Dölle method, the new method determines anisotropic elastic constants as a weighted average between single-crystal and X-ray elastic constants with weighting being done according to the relative intensities in the measured directions.

The tested samples of plate shape were made of AISI 420 (ferritic), AISI 304 (austenitic) and AISI 318LN (du-



a) Ferritic steel

b) Austenitic steel

c) Ferrite phase of duplex steel

d) Austenite phase of duplex steel

Figure 1. Determined stresses depending on external stress $'_N$ of samples with 50% reduction of thickness.

plex) type of stainless steel. The samples were cold-rolled to 1.5 mm in thickness with 0, 10, 20, 30, 40, and 50% reduction of thickness. At the end, the samples were annealed in air laboratory furnace in order to reduce residual stresses. Analysed stresses were compared with theoretical values of external stresses generated by four-point bending. Presented stresses are superposition of external stresses $_N$ and residual stresses after annealing ($_{non\text{-}bended}$). Values of $'_N$ represent theoretical value of stresses with respect to $_{non\text{-}bended}$. In ideal case, the values of should be equal to $'_N$.

The CoK radiation and *X'Pert PRO MPD* diffractometer were used to sample analysis. Texture analysis was performed on the basis of the orientation distribution function (ODF) calculated from experimental pole figures which were obtained from three diffraction lines {110}/{220}, {200}, {211} of ferrite phase and {111}/{311}, {200}, {220} of austenite phase using *MTEX* software [4]. For determination of harmonic coefficients, which are necessary to know for method of harmonic function, the *ResMat* software was used.

X'Pert PRO MPD diffractometer with manganese and chromium radiation was used to measure lattice deformations in austenite and ferrite phase, respectively. Diffraction angles 2 ^{hkl} were determined from the peaks of the diffraction lines K $_1$ of planes {311} of austenite and {211} of ferrite phase. Diffraction lines K 1 were fitted by *Pearson VII* function and *Rachinger's method* was used for separation of the diffraction lines K $_1$ and K $_2$.

The applicability of the new method of residual stress determination in textured materials was proofed for single-phase materials and for major phases of multi-phase materials to approx. 50% yield strength, see Figs. 1. However, compared with the standard sin^2 and harmonic function methods, much more accurate results were achieved. The main reason is presence of very sharp and strong texture, especially in duplex steel. Other reasons are different methods of calculation residual stresses and mainly accuracy of texture calculation.

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L6

EFFECTS OF ADVANCED LASER PROCESSING ON THE MICROSTRUCTURE AND RESIDUAL STRESSES OF H13 TOOL STEEL

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The aim of the contribution will be to describe the effects of laser processing on the microstructure and residual stresses of laser cladded H13 tool steel on the classical construct steel S355 substrate. The comparison of surface residual stresses obtained by X-ray diffraction in direction parallel (L) and perpendicular (T) to the clad is plotted in Fig. 1. Residual stresses at the beginning and the end of the clad are comparable with the experimental error. In the direction L, residual stresses exhibit slight compressive stresses along the clad. However, in the direction T, tensile residual stresses were obtained. Our research concludes that in this case of laser cladding, phase transformation and not shrinkage is likely to be a dominant effect on the formation of compressive residual stresses along the clad. Furthermore, martensitic structure and unequal concentration of alloying elements was observed on the cross-section of the clad using electron backscattering diffraction and energy-dispersive X-ray spectroscopy. These results will be compared with the data obtained by the X-ray diffraction.

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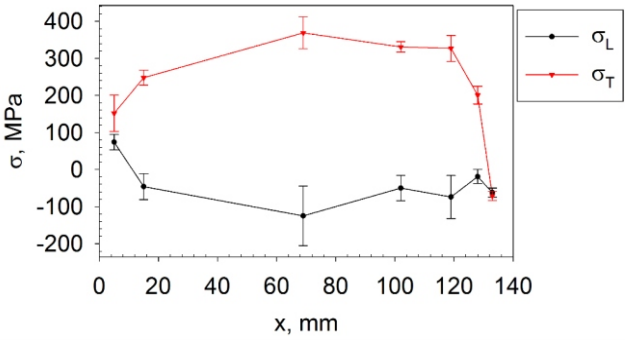

Figure 1. Surface residual stresses on the top of the clad in direction L and T along the length of laser track, where *x* is the distance from the beginning of the clad.

L7

CHARACTERIZATION OF RESIDUAL STRESS DISTRIBUTION IN STRUCTURAL MATERIALS BY NEUTRON DIFFRACTION AT CANAM INFRASTRUCTURE

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Residual stresses are phenomena that develop in all welded structures due to local heating and slight difference between the chemical composition of the welding material and welded parts. Stresses have direct influence on ductility and toughness of both weld metal and heat affected zone of the weld area, which affect the service life time of structures.

The two-axis diffractometer SPN-100 at the Center of Accelerators and Nuclear Analytical Methods (CANAM) in Řež, is an instrument dedicated to macro/micro strain scanning of polycrystalline materials. Recently, the diffractometer has been equipped with a new two-dimensional position sensitive detector (2D-PSD).

For sample positioning, in addition to standard x-y-z translation stage, a new six-axis robotic arm has been installed to allow more flexible manipulation of complex samples.

Recent neutron diffraction experimental results obtained with the upgraded tools will be presented, this include:

- Strain/stress distribution measurements in the vicinity of butt weld from spherical storage tank made of C-Mn unalloyed steel.
- Comparison of residual stresses obtained after joining thick sheets of structural steel by the laser welding and arc welding methods.
- Study of residual stresses in cold rotary swaged tungsten heavy alloy.

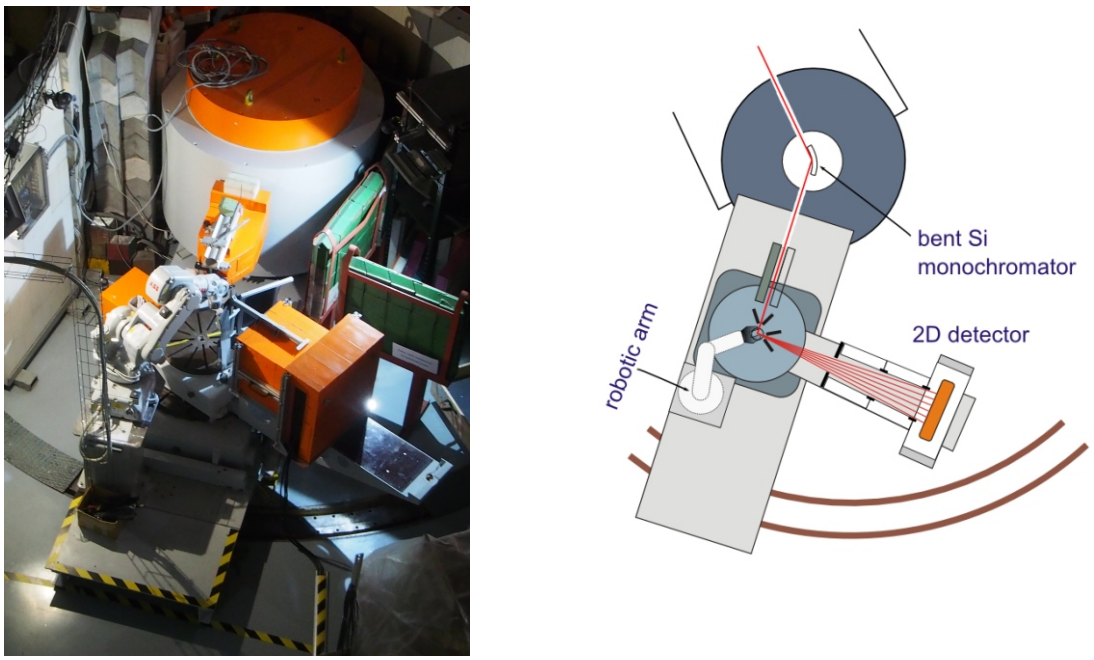

Figure 1. Layout of the neutron strain/stress scanner at the large research infrastructure Center of Accelerators and Nuclear Analytical Methods (CANAM).

L8

STRUCTURE EVALUATION OF DUAL PHASE STEELS BY EBSD METHOD

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For the development of new steel grades, it is necessary to know the microstructure and texture of rolled steel substrate because every technological process forms specific microstructure and texture of the material. Laboratory diffraction methods XRD (X-ray diffraction) and EBSD (Electron Back-Scattered Diffraction) are suitable for the knowledge of microstructure and texture. Both methods describe the texture in the form of pole figures, orientation distribution function (ODF) and selected texture fibers. Texture is very important for the materials with high anisotropy. For the dual phase (DP) steels the microstructure is more important. The content of retained austenite and martensite fraction in the ferrite are parameters which define the steel properties. For the DP steels with hardness of 1000 MPa the amount of austenite was measured at 6%. The most precise and efficient measurement of austenite is by means of XRD. The sample is polished and for the inhibition of anisotropy, the sample rotates during the measurement. Determination of austenite content was not done precisely by means of EBSD as the measurement is highly dependent on the perfect sample preparation for the measurement.

However, EBSD method provides many advantages which are not provided by classical optical microscopy methods of microstructure investigation. This includes many parameters, i.e. image quality (IQ) map, inverse pole figure (IPF), grain average misorientation (GAM), kernel average misorientation (KAM), geometrically necessary dislocations (GND), grain size and others. All these parameters comprehensively describe the microstructure of DP steels. IQ parameter is very important for the determination of martensite fraction. This parameter makes provision for distortion of the lattice, therefore it is very suitable to determine the martensite fraction, what is used by many authors [1]. This method was also used by K. Radwanski [2] for the determination of the deformed ferrite fraction. The martensite content can be also calculated from fitted XRD pattern by TOPAS software which uses Rietveld method of fitting. It is necessary to enter the structures – ferrite, martensite or austenite.

Both parameters KAM and GND include information about microtensions and disorders in the grain which are expression of increased content of low-angle boundaries. Increased amount of misorientation of 0° – 5° is in general mainly observed for grain boundaries and in martensitic grains. By entering the lattice parameter and slip system it is possible to calculate the value of geometric dislocation density using GND [3]. It was found that average value of geometric dislocation density increases with the increased hardness of DP steels, see Fig. 1.

Calculation of several physical properties is possible in the software used for evaluation of the EBSD data, such as OIM or Channel5. Using these programs, it is possible to form the maps or distribution separations of Taylor

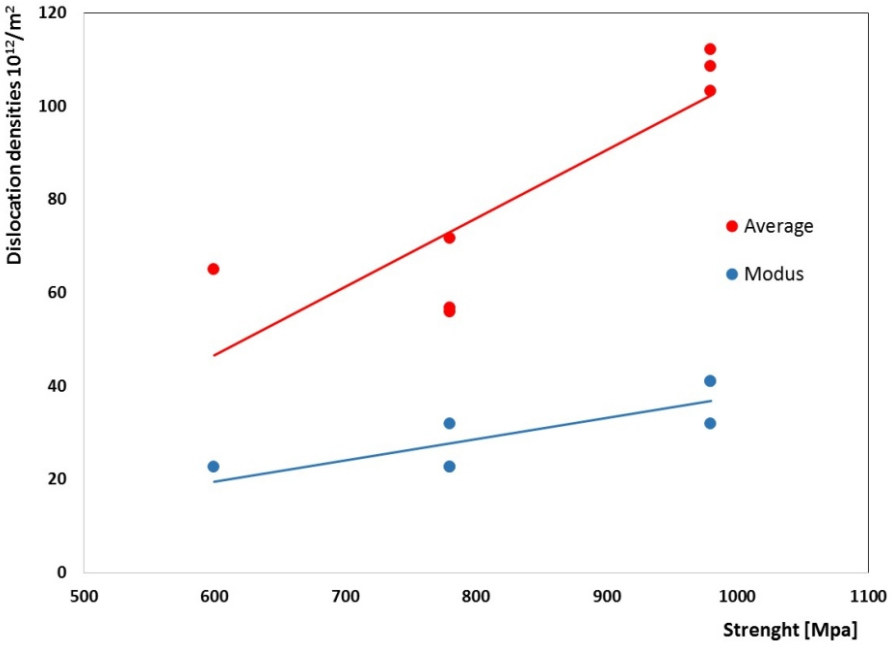

Figure 1. The effect of GND (geometrically necessary dislocations) on the strength of DP steel.

and Schmidt factors. However, these programs are not able to determine the R – Langford factor which is very suitable for practical metallurgy. This model parameter is still possible to calculate by popLA software from XRD measurements.

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