s1.m1.o3 **Refinement of Modulated Structures against Powder Diffraction Data with JANA.** M. Wunschel, R.E. Dinnebier, S. van Smaalen, *Laboratory of Crystallography, University of Bayreuth, 95440 Bayreuth, Germany*. M. Dusek, V. Petricek, *Institute of Physics, Academy of Sciences of the Czech Republic, Cukrovarnicka 10, 162 53 Praha, Czech Republic.*
Keywords: JANA, structure refinement, power diffraction data.

JANA[1] is a software package for structure refinement of regular, modulated and composite structures against single crystal data. In this contribution we present a new version of Jana, that can handle data from powder diffraction.

The motivation for this work was to make available for the analysis of powder diffraction data, the many features that are contained in JANA (e.g. modulated and composite structures, non-crystallographic site symmetry, TLS-refinement, many types of restrictions and constraints, any type of Fourier map).

JANA has been extended by a module for full profile analysis of powder diffraction data. Both Le Bail fits[2] and Rietveld refinements[3] can be performed. The recent developments concerning the descriptions of the peak profiles, like the correction for axial divergence[4] and anisotropic peak broadening[5], have been incorporated. The quality of the fit can be analyzed using an interactive graphics system with extensive features.

Several applications of the powder module will be discussed. The incommensurately modulated structure of NbTe4 is refined in (3+1)-dimensional superspace against x-ray powder diffraction data, using displacive modulations for Niobium and Tellurium atoms. The structure of $C_{60}CO$ is refined against synchrotron powder data, using restrictions given by the non-crystallographic icosahedral symmetry of the C_{60} molecule.

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s1.m1.o4 **The crystal geometry of modulation waves and the thermal expansion anomalies in IC phases**. V. Shekhtman, B. Bagautdinov, *Institute of Solid State Physics RAS, 142432, Chernogolovka, RUSSIA*
Keywords: incommensurate phases, thermal expansion, invar-effect

The paper is devoted to the XRD *in situ* investigations of phase transformations into the modulated incommensurate structure. It was established on series of experiments that the transition into IC state is accompanied by specific features in temperature behavior of definite interplanar spacings. For the first time some unusual result was demonstrated by cooling of single crystals of proustite Ag_3AsS_3. Between temperatures 60 K and 48 K, when the satellite reflections of IC phase were detected, the hexagonal parameter "*a*" not changed, i.e. in certain directions the thermal expansion coefficient (TEC) fall to zero. This phenomenon, called as an invar effect, was confirmed at 220-193 K on monoclinic crystals of $Sn_2P_2Se_6$ and was used also for determinations of IC-phase region on diagram $Sn_2P_2Se_6 - Sn_2P_2S_6$. The similar examples were obtained also by investigations of phase transitions from para− into ferro−electric phases in orthorhombic crystals of thiourea $CS(NH_2)_2$ at 202-193 K and in rhombohedral $Cs_3Sb_2I_9$ at 78-72 K. For orthorhombic compound Cs_2ZnJ_4 the invar effect is observed on reflection (001) throught the range 120-97 K of commensurate and incommensurate modulations.

The nature of this interesting phenomenon remains rather unclear. Therefore it could be useful to compare the crystallographic characteristics of presented various compounds with corresponding directions of invar-effect. The consequence data were analyzed are presented in this paper. To put the main result in crystallographic terms, the wave vector in all cases turn out as the zone axis for zero-TEC plane. Accordingly, the direction of wave vector in real space for each crystal determines the zone axis J_{mnp} to which the atomic displacements (polarization) plane is related. The model of observed anomalies can be based on the competition between static displacements of atoms under modu-lations and their anharmonic thermal vibrations. The possible explanation consider also the compensation of thermal elongation by changing of bonds tilt. The analysis of the TEC features could be used for reliable determination of atomic displacement parameters in the modulation waves.