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Gels of nominal compositions $(\text{Mg}_{1-x}\text{Co}_x)_2\text{Al}_4\text{Si}_5\text{O}_{18}$ were obtained by the sol-gel method from metallic salts (nitrates) and colloidal silica. The dried gels were melted and the amorphous precursor was annealed to crystallized monophasic cordierite solid solutions. A hexagonal cordierite (indialite) [1] and orthorhombic cordierite (iolite) [2] has been studied by several authors and are very similar; both are built of six-component rings of tetrahedral Si-Al sites perpendicular to c-axis. All these cordierites have several interesting ceramic and optical properties.

X-ray powder diffraction using Cu K α radiation and scanning electron microscopy were used to follow the structural and textural evolution from gels to the final synthetic orthorhombic cordierite solid solutions. Rietveld refinement was started using Cccm space group and structure parameters derived from Terada et al. using the FullProf program [3]. The results confirm the orthorhombic crystal structure and the solid solution formation in the whole range of compositions of these Mg-Co cordierites.

The non-periodicity of nanocrystalline materials can be analyzed using the diffraction line broadening of X-ray diffraction patterns. There are several approaches to obtain microstructural information (crystallite shapes and size distribution, microstrain). We have used an integral breadth method [4], a Fourier method [5] and a statistical method for analysing polycrystalline materials [6]. In all cases the standard line profiles for instrumental line broadening were obtained from LaB₆ (SRM 660a). On average, crystallite size of these orthorhombic cordierite solid solutions is rather similar for different metal contents, but microstrain diminishes when cobalt content increases.

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In-situ X-ray diffraction study on the decomposition kinetics in bearing steels

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We have monitored in-situ the austenite transformation kinetics in high-carbon bearing steels undergoing creep phenomena by performing time-dependent X-ray diffraction experiments under tensile load at elevated temperatures using a 70 keV synchrotron X-ray micro-beam. Bearings are nowadays used as key precision components in a broad scope of machinery and gearboxes. The commonly used high-carbon SAE 52100 steel provides the required combination of

high strength and resistance to fatigue to produce bearings with an acceptable lifetime. The room-temperature microstructure of these steels consists of martensite, metastable austenite (10-25 vol.%) and a small amount of carbides (< 5 vol.%). Small dimensional changes in the bearing during operation influences its function and thereby that of the machine in which it operates. One key process that critically affects the bearing dimensions during operation is the decomposition of the metastable austenite phase at elevated temperatures. The austenite face-centred cubic (fcc) structure transforms into the bainite body-centred cubic structure, together with the appearance of very fine nm-sized precipitates.

The combined use of the high-energy X-rays available at the ESRF and our dedicated micro-tensile stage with heating capabilities has allowed us to monitor in-situ the austenite decomposition in the bulk microstructure as a function of time for different combinations of temperature and applied tensile stress. The measurements were performed by continuously recording the diffraction patterns on a two-dimensional detector placed behind the sample as a function of time at selected temperatures in the range of 476-504 K for an applied tensile stress of: $\sigma = 0$ and $\sigma = 295$ MPa. This combination of temperature and stress mimics the operating conditions of the bearings during its lifetime. The available optics at the ID15A beam line of the ESRF permits to reduce the beam dimensions down to $5 \times 25 \mu\text{m}^2$. This has allowed us to study both the average behaviour of the metastable austenite phase and the transformation kinetics of individual austenite grains. The observed transformation behavior has been correlated to the change in sample dimensions that are recorded simultaneously during the in-situ diffraction experiment. The Rietveld analysis of the powder diffraction data reveals that the time to completely transform the metastable austenite reduces from 6 to 3 hours when increasing the temperature from 476 to 489 K. The presence of the applied tensile stress of 295 MPa reduces the activation energy from 138 to 104 kJ/mol. We have also observed a continuous decrease in the austenite lattice parameter while keeping the sample at a constant temperature. This effect is ascribed to the depletion in carbon of the remaining austenite grains resulting from the formation of iron carbides. The results of our in-situ diffraction study constitute a key input to control the austenite stability in high-carbon steels during the industrial processing of the material, in order to keep the changes in the bearing dimensions within the required limits during its lifetime.

Keywords: steel, creep, diffraction

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A multidisciplinary study of the resolution of hydratropic acid by ephedrine

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Following the introduction of regulatory guidelines for the production of stereoisomeric drugs in 1992, the number of drugs marketed as single enantiomers has increased considerably [1]. Classical resolution is often used to obtain enantiopure compounds by the pharmaceutical industry. However, despite its importance, the choice of chiral acid/base to use as a resolving agent is still mostly made on a trial and error basis. An understanding of the relationship between the crystal structure of the diastereomeric salt pairs, their relative energies and crystallization behaviour, would lead to improvements in the separation process.

In the present study we have performed an experimental and