The high-temperature superconductor (Bi,Pb)$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ (Bi2223) is one of the most promising materials in view of low-loss power applications. Its manufacture in form of flexible wires or tapes suited for the design of cables, requires a metal sheath in order to stabilise mechanically the thin strands of superconducting ceramic. In the production process known as “power-in-tube”, a mixture of precursor powders is packed into a Ag tube, which is then mechanically deformed to the required dimensions. The precursor powders do not consist in Bi2223 but in a mixture of other phases instead. After deformation of the metal/precursor powders composite, the wire/tape is heat treated at high temperature in order to convert the precursor phases into Bi2223. The conversion should however not be completed in a single heat treatment. To reach high critical current densities, an additional mechanical densification must be performed in order to increase to ceramic density. This step is then followed by a second heat treatment, during which the full conversion to Bi2223 is accomplished.

Owing to its layered structure, the electrical transport properties of the Bi2223 phase are highly anisotropic. It is therefore of crucial importance to engineer ceramic cores with a high degree of preferential orientation in order to maximise the critical current density of the composite tapes. This is usually achieved in the following way. One of the precursor phases is (Bi,Pb)$_2$Sr$_2$CaCu$_2$O$_8$ (Bi2212), which has a lamellar structure. During the initial mechanical deformation, the Bi2212 grains acquire a moderate degree of preferential orientation. The phase transformation process occurring at high-temperature involves the slow decomposition of Bi2212. The Bi2223 phase that forms over several hours, preferentially nucleates on top of the Bi2212 plate-like grains coexisting with it during the first 15 – 20 hours of heat treatment. The Bi2223 phase having also a lamellar habit, inherit the average texture of Bi2212. It is therefore crucial to improve the texture of Bi2212 in order to optimise the preferential orientation of Bi2223.

Studies of such ceramic-metal composites can be advantageously performed by means of high-energy x-ray diffraction. A 100keV beam penetrates through the Ag sheath and enables non-destructive studies in transmission geometry. In-situ investigations of the phase transformation and microstructure evolution occurring during the initial stage of heat treatment were performed on beamline BW5, using a 2-dimensional detector plate [1]. The diffraction rings, integrated using the fit2d software [2], yielded the time evolution of both texture and phase content of the Bi2212 and Bi2223 phases. The influence of the heating rate used for reaching the sintering temperature on microstructure and texture evolution has been seldom studied in previous investigations. In the present contribution, we present some new results that may have important implications for tape processing.

Short pieces of tapes were heat treated in a flow of 8.5% oxygen (rest argon) to 830°C with various heating rates ranging from 1K/min to 4K/min. Such heating rates are typical for tape processing schedules. The evolution of the Bi2212 texture during both the end of the heating ramp and the first 13h of sintering are reported in Figure 1 for heating rates of 1K/min and 4K/min. It clearly appears that the average texture of the phase improves from 750°C, nearly independently of the heating rate. This feature can be related to the grain growth occurring from the same temperature range during the heating ramp [3].

At the sintering temperature however, varying the heating rate induces significant differences in the kinetics of texturing. As evidenced in Figure 1, during the first few hours of sintering, low heating rates result in a much slower evolution of preferential orientation. After 6 to 8 hours, the degree of
preferential orientation becomes independent of the heating rate again. At this stage, about 20% phase transformation to Bi2223 occurred. We also mapped the texture evolution of the Bi-2223 phase as soon as the intensity of relevant diffraction rings was high enough. It was found to follow a similar trend.

\[ FWHM \ [^\circ] \]

**Figure 1:** Evolution of the Bi2212 phase texture (FWHM of the (117) reflection) during the heat treatment. The data are collected on a single-filament tape. Filled circles: heating rate 1K/min, open circles: heating rate 4K/min.

It thus appear that low heating rates have to be associated to long enough initial heat treatments in order to optimise the texture of the ceramic core. In fact, the degree of texture reached at this stage sets a limit to the texture achievable at the end of the final heat treatment, because the increase of ceramic density resulting from the intermediate densification step hampers further improvements [4].

One of the main problems occurring during the manufacture of long lengths of tapes is the formation of gas bubbles in the tape during the initial heat treatment. These result in extended defects that can not be healed during the final heat treatment. The use of low heating rates has been put forward in order to avoid this problem [5,6]. Our present findings demonstrate that, in such a case, one should also ensure that the first heat treatment is not too short.

**References**