ENERGY STUDY OF CU-NBC NANOCOMPOSITE PRODUCED VIA MECHANICAL ALLOYING

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Copper based nanocomposite powders reinforced *in situ* with NbC were synthesised by mechanical alloying. Mixtures corresponding to a nominal content of 20 vol. % NbC were produced from powders of Cu (99.9% purity; particle size 44 μm<d<149 μm), Nb (99% purity; average particle size 65 μm) and synthetic graphite (99.9995% purity; average particle size 74 μm). The milling was performed in two steps. Firstly, a mixture of Cu and graphite powders was milled during 4 hours with a rotation speed of 400 rpm to produce a Cu-C pre-alloyed powder. This step has two main objectives, to deoxidise Cu and to disperse graphite into the copper matrix. In the second step (*in situ* NbC synthesis) Nb was added to the mixture and milling continued for different times, namely, 1, 2, 4, 8, 16 and 32 hours. In this step three different values of rotation speed, ω_r, of 300, 350 and 400 rpm were used. A detailed account of the processing methods was reported elsewhere .

As-milled powder was characterized by X-Ray Diffraction. XRD experiments were performed with a Rigaku Geigerflex difractometer fitted with a graphite monochromator, using CuK_α radiation. Scanning speed 0.006 °/min (2θ) was used for high resolution scanning of (111) Cu and (111) NbC peaks (in the 32-54 ° 2θ range). The volume fraction of NbC, f_{NbC}, was calculated from the ratio of (111) NbC and (111) Cu peaks, I_{NbC}/I_{Cu}. Kinetics was described by the equation:

$$x = a(1 - \exp(-kt)) \quad (1),$$

where x is the volume fraction transformed at the time t, k is energy dependent rate constant and a is a fitting parameter related with the concentration of reactants.

The volume fraction of the NbC *versus* milling time for ω_r of 300, 350 and 400 rpm is presented in Figure 1 (a) as well as the fitted curve with equation (1). Table 1 shows the values of k and a obtained from the fitting of experimental data and, the milling time needed to achieve the maximum proportion of NbC ($t_{f_{NbC}}$ max.) for the three values of rotation speed used.

Results indicate that the formation of NbC is dependent of the level of energy used in the milling process. For a rotation speed of 350 rpm the milling time required to obtain 94% of de transformation is about 4 hours whereas the time to achieve about 93% of transformation for 300 rpm is approximately 16 hours. For ω_r=400 rpm the maximum of transformation 88% is obtained after 8 hours of milling. These results are in good agreement with the rate constants determined for the three cases, i.e., k_{350} is higher than the other two rate constants, see Table 1. The energy corresponding to ω_r=400 rpm is too high while for ω_r=300 rpm the energy is too low. These important results allowed the drawing of a simple milling map for the Cu-20NbC system, as shown in Figure 1 (b). Therefore, the energy corresponding to a rotation speed of 350 rpm appears to be close to the optimised conditions to obtain the maximum proportion of NbC in a short period of milling. The existence of k at intermediate rotation speed may be related to the disturbance of NbC synthesis mechanism at higher ω_r.

References:

Figures:

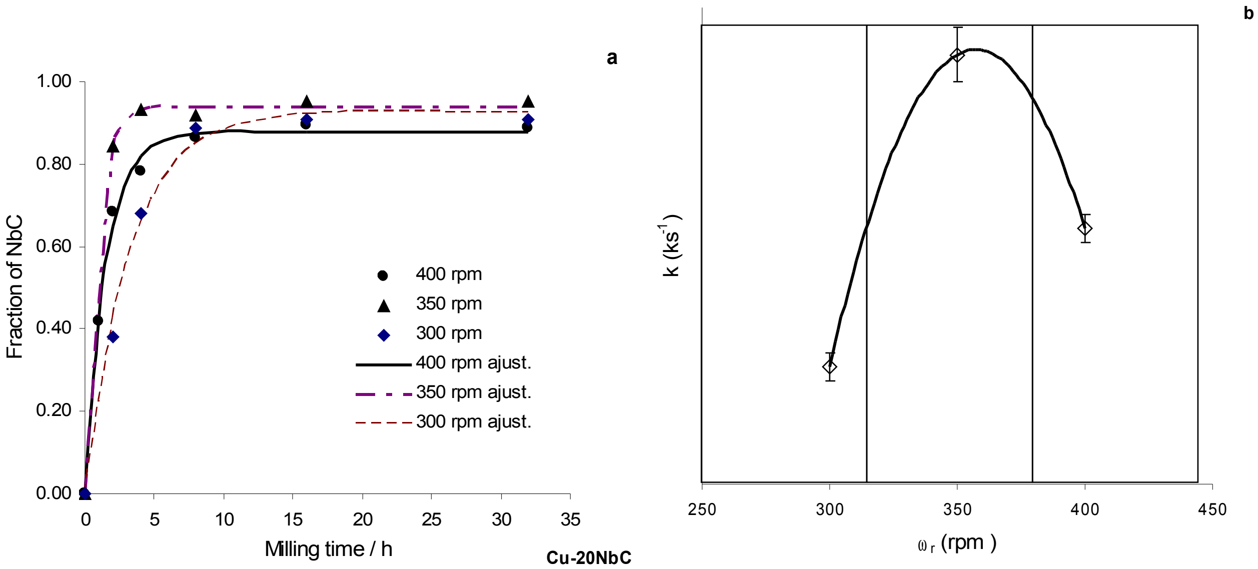

Figure 1: (a) Volume fraction of NbC vs. milling time for different rotation speeds; (b) rate constant, k, for ω_r of 300, 350 and 400 rpm.

Table 1: Kinetics parameters obtained, from XRD, for the Cu-20NbC powders milled with a rotation speed of 300, 350 and 400 rpm. The milling time to achieve the maximum of transformation is also shown.

Sample Id.	k (ks^{-1})	a	$t_{f_{NbC}}$ max (h)
Cu-20NbC; 400 rpm	0,19 ± 0,01	0,88 ± 0,01	8
Cu-20NbC; 350 rpm	0,31 ± 0,02	0,94 ± 0,01	4
Cu-20NbC; 300 rpm	0,09 ± 0,01	0,93 ± 0,02	16