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# Regular Article Time evolution of mechanical amorphization: A kinetic model

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#### ARTICLE INFO

### ABSTRACT

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Keywords: Mechanical alloying or grinding Amorphous materials Mechanical amorphization A model is proposed to describe the evolution of the amorphization process driven by mechanical grinding. This model considers the interface controlled growth of the disordered regions at the boundaries between the crystallites (unlike the classical nucleation and growth theory, the growing particles are thus concave which affects the kinetic equation). The validity of this model has been tested for the amorphization process of mechanically alloyed Fe-Nb-B system at different frequencies. Predictions of the model are in agreement with the experimental results and values of the linear growth rate and the thickness of the starting disordered layer are obtained. © 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Since the pioneer developments in the early 80's of amorphous systems produced via milling by Yermakov et al. [1] and Koch et al. [2], mechanical alloying and/or grinding has been shown as a suitable technique to produce amorphous systems even in a wider composition range than that obtained by rapid quenching [3]. However, although mechanical amorphization was achieved >30 years ago, the kinetics of how this amorphization process occurs has not been elucidated yet. Excellent reviews on ball milling and mechanical alloying can be found in the literature. Suryanarayana [4] dedicates a chapter to mechanical amorphization, describing the different attempts to characterize the thermodynamics of amorphization via milling. In general, the conditions described are those for the amorphous phase to be more thermodynamically stable than the crystalline counterpart with high concentration of defects. More recently, Balaz et al. collected the literature on transformation kinetics during milling [5]. They describe the quantitative results of Delogu et al. [6,7] based on a statistical interpretation of the progress of transformation in the milled powder. This idea was applied by Delogu and Cocco [8] to interpret the kinetics of mechanical amorphization (as well as other transformations [9,10,11]) based on the number of impacts a powder particle needs to be amorphized. In the case of planetary mills, in the frame of the early descriptions of dynamics of planetary mills developed by Abdellaui and Gaffet [12] and Magini et al. [13], it was shown that the power released to the powder during milling is proportional to the cube of the frequency of the main disc,  $\Omega$ , which allows for a continuous description [14] unlike the discrete description based on number of collisions. However, the focus of this paper is to propose a nucleation and growth model to describe the time dependence of the amorphous fraction. Concerning the time evolution of the amorphous

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http://dx.doi.org/10.1016/j.scriptamat.2016.12.019 1359-6462/© 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved. fraction during milling, Delogu et al. [15] describe the sigmoidal-like curves assuming that the transformation rate is proportional to the surface between the reagents. Other authors just report a qualitative description [16], phenomelogical exponential laws [17] or directly apply the classical nucleation and growth theory [18,19,20] (summarized in the Johnson-Mehl-Avrami-Kolmogorov (JMAK) theory [21, 22,23]).

Analyzing the five postulates required for JMAK theory we can find some clues about the suitability of JMAK theory to describe mechanical amorphization [24]:

- 1 an initial phase progressively and completely transforms to a product one,
- 2 the volume of any transformed region is much smaller than the whole volume of the system,
- 3 the nucleation is random,
- 4 convex shape of the growing phase units, and
- 5 the linear growth rate can be expressed as a product of a time dependent function and a direction dependent function.

Postulates 1, 2 and 5 are followed by mechanical amorphization as the amorphous phase can completely substitute the supersaturated solid solution previously formed during milling, the regions to be amorphized correspond to the precursor crystals of the supersaturated solid solution (generally of nanometric size), and it is expected that the amorphous-crystalline boundary advances as an interface controlled process due to the low temperature at which the process occurs and that, once the supersaturated solid solution is formed, diffusion should not be the determinant mechanism.

However, postulates 3 and 4 are not fulfilled by mechanical amorphization. On the one hand, nucleation of the amorphous regions is not random but nuclei for the amorphous phase are already located in the disordered boundary regions between the nanocrystals. On the





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other hand, the thickening of these boundary regions leading to transform a nanocrystal to amorphous phase should present a concave shape instead of the convex one required in JMAK theory.

In order to take into account these deviations, we have developed a simple model describing the growth of the amorphous boundary region to the inner volume of the nanocrystal, transforming it to an amorphous phase. The predictions of this model have been compared with experimental results on the amorphization of mechanically alloyed  $Fe_{75}Nb_{10}B_{15}$  alloy.

 $\rm Fe_{75}Nb_{10}B_{15}$  alloy was produced by mechanical alloying pure powders at two different revolution frequencies (150 and 350 rpm) in a Fritsch Pulverisette Vario 4 planetary mill. As it was shown earlier [14], the power released to the powder during milling scales as a function of the cube of the frequency and this allows us to represent both milling experiments in a common axis using the equivalent time,  $t_{eq}$ , description:

$$t_{eq} = t \left(\frac{\Omega}{\Omega_0}\right)^3 \tag{1}$$

where *t* is the time,  $\Omega$  is the frequency of the experiment and  $\Omega_0$  is the frequency to which the time is going to be rescaled (in this study  $\Omega_0 = 150$  rpm). This is in agreement with the results of Delogu et al. [15], who found that the experimental amorphization curves can be rescaled once the dose is known. In our interpretation, the dose, equal to the energy released to the powder, should be proportional to the product of milling time and  $\Omega^3$ .

The fraction of the amorphous phase developed as a function of the milling time was estimated from X-ray diffraction (using Cu K $\alpha$  radiation) and from Mössbauer spectrometry (using a <sup>57</sup>Co(Rh) source in transmission geometry in a Wissel spectrometer). Fig. 1 shows the



**Fig. 1.** XRD patterns and Mössbauer spectra for selected equivalent times ( $\Omega_0 = 150$  rpm). Red numbers in parenthesis indicate the experimental time for those results obtained at 350 rpm. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

XRD and Mössbauer spectra for some selected equivalent times ( $\Omega_0 = 150$  rpm). Further details about the milling parameters, Mössbauer fitting and microstructure analysis can be found elsewhere [25].

Fig. 2 shows the amorphous fraction as a function of the equivalent milling time. As it can be observed in the inset of Fig. 2, Mössbauer data are much more sensitive than XRD ones to detect small fractions of amorphous phase. Therefore, the area fraction of the paramagnetic contribution (Fig. 2) were used to estimate the amorphous phase fraction, X, and to test the theoretical predictions derived from this study.

The model we propose here is based on the presence of already disordered regions surrounding each nanocrystal (intergrain regions), which will act as the germens for the amorphous phase. For simplicity, we will consider a cubic region of length  $D_0$ . We will assume that the amorphous layer thickens with a linear rate L(t) = Gt (interface controlled growth), where *t* is the time and *G* is the linear growth rate. It is easy to estimate the transformed fraction for a certain time *t* from the difference between the relative volumes of the inner cube (remaining crystalline phase) and the complete cube:

$$X * (t) = \frac{D_0^3 - (D_0 - 2L(t))^3}{D_0^3} = \frac{D_0^3 - (D_0 - 2[L_0 + G(t - t_0)])^3}{D_0^3}$$
(2)

where  $L_0$  is the initial thickness of the disordered region surrounding the crystal and  $t_0$  is the induction time of the amorphization process. It is worth mentioning that Eq. (2) holds for any regular 3D object which can be defined by a length (i.e. the diameter of a sphere). On the other hand, although Eq. (2) is developed for an interface controlled process, it is easily extended to diffusion controlled processes just taking  $L(t) = L_0 + \sqrt{G(t-t_0)}$ .

However, the fraction described by Eq. (2) does not consider the presence of competitor amorphous regions, i.e. it is equivalent to the extended transformed fraction described in JMAK theory. Following JMAK theory [22], this extended transformed fraction  $X^*$  is related to the actual transformed fraction X as:

$$\frac{dX}{dX^*} = 1 - X \tag{3}$$

and integrating from  $X(t_0) = X^*(t_0) = X_0 < <1$  can be also written as:

$$-\ln(1-X) + \ln(1-X_0) = X^* - X_0 \tag{4}$$



**Fig. 2.** Amorphous fraction obtained from Mössbauer data as a function of the equivalent milling time. Hollow symbols were experimentally obtained at 350 rpm and the time was rescaled to the corresponding equivalent one at  $\Omega_0 = 150$  rpm. Line corresponds to the fitted curve using the model developed in this study. Inset shows the higher sensitivity to detect small amorphous fraction from Mössbauer data with respect to those from XRD data.

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