



Intensification of a high-temperature phase transformation by ball milling

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ABSTRACT

This work focuses on the allotropic phase transformation occurring in Co powders at high temperature. First, we studied the phase transformation kinetics in Co powders that are exposed to a temperature of 750 K while being stirred. Then, we investigated the phase transformation kinetics in Co powders that are ball milled at the same temperature. We observe that the mechanical processing induces a significant intensification of the phase transformation, which we relate to the effects of individual impacts occurring during the mechanical processing. We developed an analytical kinetic model that provides a reliable description of the phase transformation kinetics while accounting for the discontinuous nature of ball milling. We not only explain the observed intensification on a sound phenomenological basis, but also define an intensification index to measure the extent of intensification. The results can be extended to other cases where ball milling intensifies a transformation process.

It is well known that, at temperatures above 700 K, Co undergoes a phase transformation from a phase with close-packed hexagonal (hcp) structure to a phase with face-centred cubic (fcc) structure [1–6]. Associated with a latent heat of ca. 500 J mol⁻¹, the phase transformation is sluggish and characterized by significant reversibility and hysteresis. The change of crystalline structure occurs through a diffusionless mechanism based on the cooperative realignment of Co atoms. For these reasons, the hcp-to-fcc phase transition can be classified as a first-order martensitic phase transformation [1–6].

Not only heating, but also the severe deformation of bulk samples and the ball-milling (BM) of powders can induce the phase transformation [7–10]. The case of BM is particularly interesting in this regard. The allotropic phase change occurring in Co powders subjected to BM have been related to the formation of stacking faults mediated by crystallographic slip and twinning in a disordered hcp structure with distorted unit cells [7–10]. The appearance of the fcc phase does not seem to correlate with the grain size refinement or the local temperature rise caused by impacts [7–10]. Impurity contamination has been also convincingly ruled out [7–10].

While these details provide valuable tiles of the overall mechanistic puzzle displaying on the atomic level, much less is known about the phase transformation kinetics unfolding on a coarser scale. This has far-reaching consequences on the proper understanding of the way in which the discontinuous nature of BM gives shape to the experimental kinetic curve, which usually results in misleading interpretations of the

available kinetic evidence [11–13].

The fact that the same considerations apply to the generality of physical and chemical transformations induced by BM gives a glimpse of the challenge. At the same time, it makes clear that the tiniest progress in the understanding of the phase transformation behaviour of Co powders induced by BM can have important implications for the entire field of study, especially now that mechanochemistry, and mechanical processing in general, have regained momentum [14–19].

In this work, we show exactly how BM affects the apparent phase transformation kinetics. To this aim, we studied, first, the phase transformation undergone by loose Co powders stirred inside the moving reactor of a ball mill kept at the temperature of ca. 750 K, which we call dynamic annealing (DA). Then, we investigated the phase transformation under BM conditions at the same temperature. Not only do we demonstrate that BM intensifies the phase transformation that occurs under DA conditions, but we also relate the observed rate enhancement to the effects of individual impacts.

We performed DA and BM experiments using high-purity Co powders with particle size below 10 μm, which were annealed for 24 h at 200 °C under a flux of Ar and H₂ to eliminate humidity and surface oxides. Annealed powders, and subsequently processed ones, were stored and handled under Ar atmosphere with H₂O and O₂ contents below 2 ppm.

DA experiments were run placing 10 g of Co powder inside a hardened-steel cylindrical reactor with flat bases and a total volume of about 60 cm³. The reactor temperature was controlled electronically

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using a handcrafted, electrically powered ceramic band heater wrapped around the reactor in combination with a thermocouple attached to one of the reactor ends. We secured the reactor to the clamp assembly of a Spex Mixer/Mill 8000, having the foresight to interpose thin cylinders in refractory material between the two. The reactor temperature was kept at 750 K and the ball mill was operated at a frequency of 18 Hz.

We run BM experiments under the same conditions, adding a stainless-steel ball of 12 g to the powders. We characterized the BM conditions by detecting the ball impacts on the reactor bases and side wall with a piezoelectric sensor. The piezoelectric signals were recorded and suitably analyzed. We also numerically simulated the ball motion inside the reactor. We estimated the impact frequency associated with the real processing conditions by comparing experimental and numerical outcomes.

We used X-ray diffraction (XRD) and electron microscopy to monitor the phase transformation. Accordingly, DA and BM were interrupted every 10 min and the reactor rapidly cooled using powerful jets of cold air. Then, Co powders were sampled and XRD patterns were collected using a Rigaku D/Max diffractometer equipped with Cu K_α radiation. For each time interval, we estimated the average fractions of hcp and fcc phases from XRD patterns using the Rietveld method.

Full details on materials and methods are given in Supplementary Material SM.1.

The results of the quantitative XRD analysis (see Supplementary Material SM.2) for DA and BM experiments are shown in Fig. 1a. In both cases, the fraction of fcc phase, $\chi_{fcc}(t)$, increases monotonically with time, t , tending to 1 asymptotically. The semi-logarithmic plots in Fig. 1b indicate that BM is associated with an almost perfect linearity and, thus, an exponential $\chi_{fcc}(t)$ change, whereas the data arrangement not far from linearity suggests that the exponential is still a good

approximation for the $\chi_{fcc}(t)$ time dependence resulting from DA experiments.

The $\chi_{fcc}(t)$ values characterizing BM experiments invariably surpass the corresponding ones from DA experiments. This provides the undeniable evidence that, in addition to causing powder comminution (see Supplementary Material SM.3), BM eventually induces a significant rate enhancement of the hcp-to-fcc phase transformation undergone by Co powders under DA conditions. About 18 min are needed for $\chi_{fcc}(t)$ to reach the value of 0.5 under BM conditions, while it takes ca. 36 min under DA conditions.

We can ascribe the observed rate enhancement only to the effects of the plastic deformation that Co powders experience when they are compressed at impact during BM experiments. Superposing to the phase transformation that already involves uniformly all the Co powders as a consequence of the high temperature, such effects speed up the phase transformation in the myriad of small volumes sequentially affected by impacts, finally reverberating on the global phase transformation rate.

To translate these conjectures into a rigorous explanation consistent with the experimental evidence and the inherently discontinuous nature of BM, we refer to the fact that, during individual impacts, small amounts of powder experience dynamic compaction at relatively high strain rates. The contact forces between neighbouring particles can locally exceed the Co yield point, giving rise to effective plastic deformation processes [11–13,20–26]. Small volumes \mathbf{v}^* , irregularly distributed within the total volume of compressed powder, can experience critical loading conditions (CLCs) [11–13,20–26]. In such volumes \mathbf{v}^* , the phase transformation can proceed to a greater extent than in the rest of the powder. In other words, there exists a volume \mathbf{v} , equal to the sum of volumes \mathbf{v}^* , where the phase transformation progresses abruptly due to the CLCs experienced during the impact. After such episode, the volume \mathbf{v} will return to the more gradual phase transformation behaviour induced by DA conditions until it will experience CLCs again.

For any given volume \mathbf{v} , the phase transformation comes down to a sequence of gradual progresses and sudden rises in the total amount of fcc phase. The length of the time intervals separating two consecutive CLCs in the sequence changes irregularly. This gives the impression that the $\chi_{fcc}(t)$ change is at the mercy of chance and its mathematical description is precluded. However, it is not.

Let us assume, for convenience, that impacts occur in a regular sequence where consecutive impacts are separated by time intervals Δt much shorter than the time required to complete the phase transformation. This assumption may sound implausible, but experiments and numerical simulations (see Supplementary Material SM.4) provide evidence, such as the one shown in Fig. 2, that clearly suggests that 10 g of Co powders allow the ball to undergo regular and periodic displacements between the two reactor bases with time intervals Δt between consecutive impacts of ca. 28 ms (see Supplementary Material SM.5).

Similarly, we assume that the impact duration, τ , is much shorter than Δt , which is reasonably expected [27]. Furthermore, we assume that phase transformation, both during individual impacts and the time intervals separating two of them, proceeds at a rate proportional to the total amount of residual hcp phase. Accordingly, we can write that the fraction of fcc phase after the first time interval Δt has elapsed, $\chi_{fcc}(1; 0)$, is equal to

$$\chi_{fcc}(1; 0) = k_{DA} \chi_{hcp}(0; 0) \Delta t, \quad (1)$$

where $\chi_{hcp}(0; 0)$ is the initial fraction of hcp phase, equal to 1, and k_{DA} is the apparent rate constant of the phase transformation under DA conditions. If the volume \mathbf{v} containing a fraction of fcc phase equal to $\chi_{fcc}(1; 0)$ undergoes CLCs, the fraction of fcc phase inside the volume \mathbf{v} becomes equal to

$$\chi_{fcc}(1; 1) = k_{BM} \chi_{hcp}(1; 0) \tau, \quad (2)$$

where k_{BM} is the apparent rate constant of the phase transformation

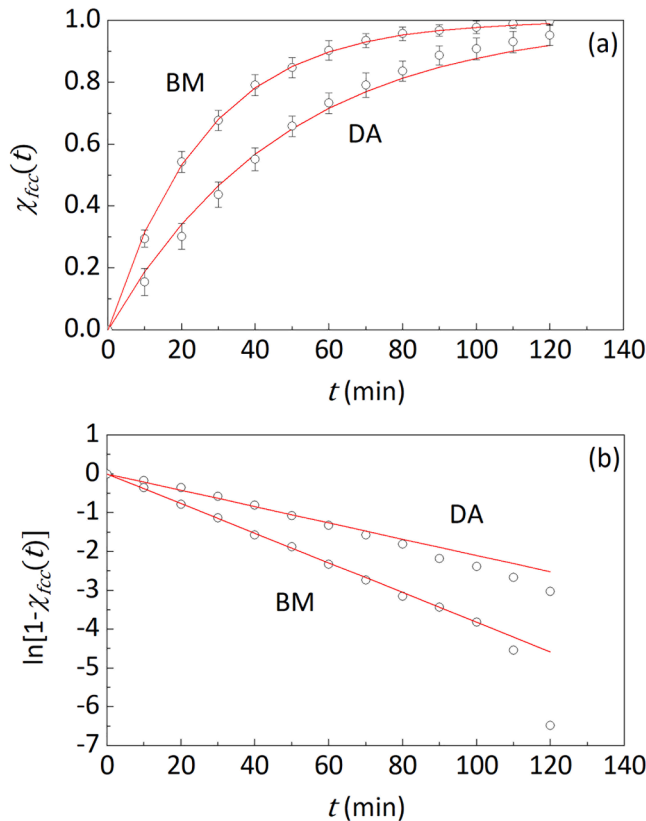


Fig. 1. (a) The fraction of fcc phase, $\chi_{fcc}(t)$, and (b) the logarithm of $1 - \chi_{fcc}(t)$, $\ln[1 - \chi_{fcc}(t)]$, as a function of time, t . Data refer to Co powders that have experienced DA and BM conditions. Best-fitted exponential curves and lines are shown.

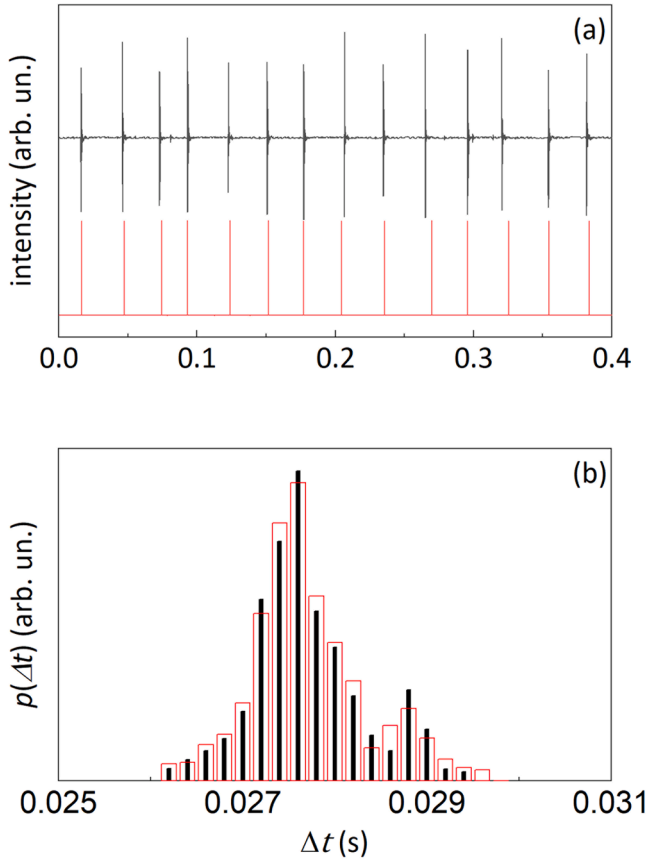


Fig. 2. Typical sequences of impacts from experiments (black line) and numerical simulation (red line). (b) Statistical distributions $p(\Delta t)$ of the time intervals between consecutive impacts obtained experimentally (black columns) and numerically (red columns).

under BM conditions, i.e. enhanced by impacts. Otherwise, if the volume \mathbf{v} containing a fraction of fcc phase equal to $\chi_{fcc}(1;0)$ does not undergo CLCs and a second time interval Δt elapses, the fraction of fcc phase changes into

$$\chi_{fcc}(2;0) = k_{DA} \chi_{hcp}(1;0) \Delta t. \quad (3)$$

All of this can be translated into the iterative scheme shown in Fig. 3, which reveals intuitively the relationship between $\chi_{fcc}(j;i)$ and the

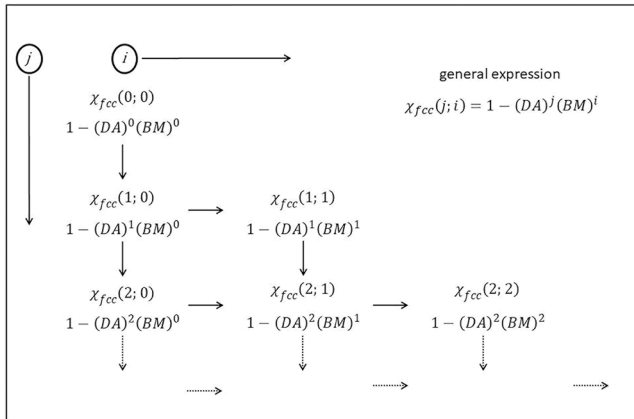


Fig. 3. A schematic description of the relationship between the number j of time intervals Δt elapsed, the number i of impacts occurred and the fraction $\chi_{fcc}(j;i)$ of fcc phase. For convenience, DA and BM are used to indicate the quantities $1 - k_{DA} \Delta t$ and $1 - k_{BM} \tau$ respectively.

numbers j and i of, respectively, time intervals Δt elapsed and CLCs experienced. Algebra leads straightforwardly to (see Supplementary Material SM.6)

$$\begin{aligned} \chi_{fcc}(j;i) &= 1 - (1 - k_{DA} \Delta t)^j (1 - k_{BM} \tau)^i \\ &\approx [1 - \exp(-k_{DA} \Delta t)] [1 - \exp(-k_{BM} \tau i)], \end{aligned} \quad (4)$$

which accounts exactly for the extent of phase transformation in a volume \mathbf{v} due to gradual progresses during the time intervals Δt or sudden rises caused by CLCs. It follows that the phase transformation kinetics under DA conditions can be described by the equation (see Supplementary Material SM.6)

$$\chi_{fcc}(j;0) = 1 - \exp(-k_{DA} \Delta t j). \quad (5)$$

It is worth noting that Eqs. (4) and 5 describe only the local kinetics in a volume \mathbf{v} . To move from local kinetics to global kinetics, which is portrayed by the kinetic datasets collected experimentally, we need to address the issues regarding the relationship between the way different volumes \mathbf{v} in a total volume \mathbf{V} of Co powders are affected by CLCs as a consequence of impacts and the overall change of the fraction of fcc phase, $\chi_{fcc}(m)$, with the total number of impacts, m , occurring during BM.

Under a few simplifying assumptions invoking the homogeneity of Co powders, the stochastic involvement of volumes \mathbf{v} in CLCs and the constancy of the fraction of powder undergoing CLCs during individual impacts, the volume fraction of powder that has undergone CLCs i times after m impacts can be expressed as (see Supplementary Material SM.7)

$$\chi_i(m) = \left[(k_{CLC} m)^i / i! \right] \exp(-k_{CLC} m), \quad (6)$$

where k_{CLC} , equal to the ratio \mathbf{v}/\mathbf{V} , represents the fraction of powder affected by CLCs during a single impact.

A glimpse at Fig. 3 indicates that the total number j of time intervals Δt elapsed when a total number of impacts m has occurred is equal exactly to m . Therefore, the total time elapsed, t , is equal to $m \Delta t$ and the total fraction of fcc phase after m impacts is given by the weighted sum

$$\chi_{fcc}(m) = \sum_{i=0}^m \chi_i(m) \chi_{fcc}(m;i), \quad (7)$$

which leads to the expression (see Supplementary Material SM.5)

$$\chi_{fcc}(m) = 1 - \exp(-k_{DA} \Delta t m) \exp(-k_{CLC} k_{BM} \tau m). \quad (8)$$

As the regular sequence of impacts indicates an approximately constant impact frequency N of ca. 35.9 Hz, Eqs. (5) and 8 can be used to best fit the experimental datasets in Fig. 1 by simply replacing m with the product $N t$. Given their exponential shape, the model curves best fit almost perfectly the dataset from BM experiments and satisfactorily well the dataset obtained under DA conditions.

More importantly, Eq. (8) accounts for the intensification of the phase transformation occurring under DA conditions consequent to the impacts occurring during BM. The contribution given by BM to the overall phase transformation can be measured by the difference between the two datasets, $\Delta \chi_{fcc}(m)$, which is plotted in Fig. 4 as a function of the number of impacts, m . It can be seen that data exhibit a non-monotonic change with a maximum approximately between 5×10^4 and 9×10^4 impacts. The equation for $\Delta \chi_{fcc}(m)$ can be readily obtained by taking advantage of Eq. (8) (see Supplementary Material SM.8). In particular,

$$\Delta \chi_{fcc}(m) = \exp(-k_{DA} \Delta t m) [1 - \exp(-k_{CLC} k_{BM} \tau m)]. \quad (9)$$

Eq. (9) best fits the experimental data to a remarkable extent with k_{DA} and $k_{CLC} k_{BM} \tau$ values of ca. $4.3 \times 10^{-4} \text{ s}^{-1}$ and 1.1×10^{-5} respectively.

Proceeding further along the same line, we note that the area between the kinetic curves obtained under BM and DA conditions, and

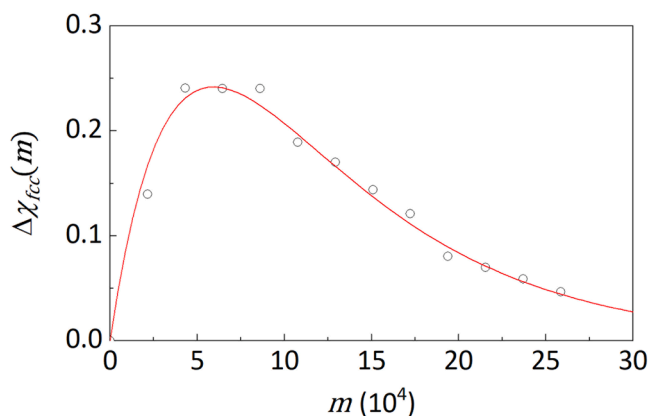


Fig. 4. The difference, $\Delta\chi_{fcc}(m)$, between the fractions of fcc phase, $\chi_{fcc}(m)$, for the phase transformations occurring under BM and DA conditions as a function of the total number of impacts, m . Best-fitted curve is shown.

shown in Fig. 1, represents a natural candidate to measure quantitatively the extent of intensification of the phase transformation due to BM. In fact, the larger the area, the more the phase transformation is intensified. Therefore, we define the intensification index

$$\Delta_{int} = \int_0^{\infty} \Delta\chi_{fcc}(m) dm, \quad (10)$$

which is equal to (see Supplementary Material SM. 6)

$$\Delta_{int} = -(k_{CLC} k_{BM} \tau + k_{DA} \Delta t)^{-1} + (k_{DA} \Delta t)^{-1}. \quad (11)$$

It is immediate to see that Δ_{int} tends to 0 when k_{BM} tends to 0, i.e. when individual impacts during BM do not result in any abrupt contribution to phase transformation, while Δ_{int} tends to infinity when k_{DA} tends to 0, i.e. when the phase transformation does not occur under DA conditions, but only consequent to BM. In the present case, Δ_{int} is equal to ca. 39.6 and we can reasonably expect that larger values are obtained increasing the intensity of BM.

In summary, we have investigated the hcp-to-fcc phase transformation occurring in loose Co powders under DA and BM conditions at high temperature. To this aim, we have developed an experimental set-up to maintain the ball mill reactor at the constant temperature of 750 K while acquiring data on the sequence of impacts occurring during BM experiments, which we subsequently compared with the corresponding outcomes of the numerical simulation of ball motion. Quantitative XRD analysis provided accurate kinetic datasets showing that BM significantly intensifies the phase transformation process taking place under DA conditions. Then, we built a kinetic model consistent with the discontinuous nature of BM and related the observed intensification to the effects of individual impacts, also measuring intensification through a suitably defined intensification index.

The exponential shape of the kinetic curves obtained under BM and DA conditions has given us the opportunity to develop a fully analytical kinetic model, but the conceptual framework can be readily extended to non-analytical case studies. In addition, we emphasize that the model can be applied to all the possible physical or chemical transformations that prove to be intensified by BM.

CRedit authorship contribution statement

Maria Carta: Writing – review & editing, Validation, Methodology, Investigation, Formal analysis. **Marta Cappai:** Writing – review & editing, Validation, Methodology, Investigation. **Giorgio Pia:** Writing – review & editing, Validation, Methodology, Investigation. **Francesco Delogu:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Project administration, Methodology,

Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.scriptamat.2026.117344.

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