

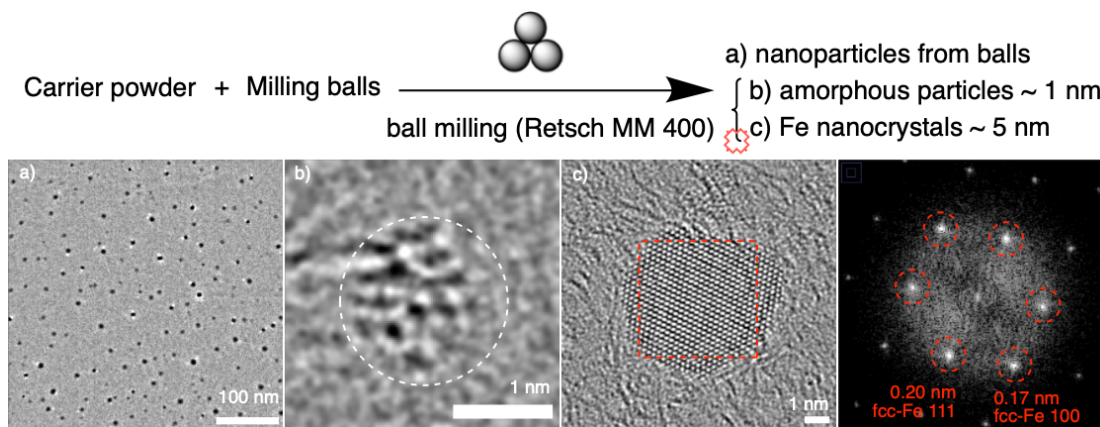
## Ball-milling causes Fe nanoparticle formation in mechanochemical processes.

(<sup>1</sup> Graduate School of Science, *The University of Tokyo*) ○Naoto Yogi,<sup>1</sup> Masaya Sakakibara,<sup>1</sup> Takayuki Nakamuro,<sup>1</sup> Eiichi Nakamura<sup>1</sup>

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Mechanochemistry has gained significant demand in recent years in the chemistry field from the perspective of green chemistry. By driving reactions through mechanical stimuli from balls, mechanochemistry often exhibits unique reactivity, making it a powerful method for the development of new reactions and the exploration of new materials. However, the potential formation of byproducts induced by mechanical stimuli has not been thoroughly evaluated to date.<sup>1</sup>

In this study, we discovered that under typical operating conditions of a mixer mill (Retsch MM400) using either pure iron or stainless-steel balls with reasonable amounts of carrier powder such as carbon materials and inorganics, amorphous particles of approximately 1 nm in size and Fe nanocrystals of about 5 nm are generated. Generally, bulk iron adopts a BCC structure and may exhibit a BCT structure depending on the amount of carbon dissolved in the lattice. It was reported that both BCC and FCC structures were observed in the bottom-up Fe nanocrystal synthesis.<sup>2,3</sup> Here, the nanoparticles observed in this study showed an FCC structure with over 95% purity, suggesting that polymorphic transformation occurs during this top-down procedure. We will discuss the details of experiments and mechanistic interpretations.



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