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Cold sintering problem in the high energy ball milling of iron-titanium powder mixtures

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TiFe intermetallic compound is well known as a material for hydrogen storage in solid form. High energy ball milling has been used to provide a better hydrogen intake in this compound. The purpose here is to mill a mixture of titanium and iron powders in order to activate the powders to further react them to produce the desired compound TiFe by heat treating the milled mixture. Activation was varied by increasing the milling time in a SPEX mill (starting from 1 hour), keeping constant the other milling parameters like ball to powder ratio, ball diameter and wt.% of process control agent. Some unexpected results were obtained with milling time higher than 1 hour. A kind of sintering occurred, which impaired the milling action. This problem, not reported before in the literature, was solved only by increasing the quantity of the process control agent. SEM, X-ray diffraction and Mossbauer spectroscopy analyses were carried out to explain this behavior.