

Accepted Manuscript

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PII: S0925-8388(18)31254-4

DOI: [10.1016/j.jallcom.2018.03.377](https://doi.org/10.1016/j.jallcom.2018.03.377)

Reference: JALCOM 45607

To appear in: *Journal of Alloys and Compounds*

Received Date: 11 December 2017

Revised Date: 10 March 2018

Accepted Date: 28 March 2018

Please cite this article as: F. Zhang, Y. Liu, J. Li, R. Wang, Ultrafine nanocrystalline NdFeB prepared by cryomilling with HDDR process, *Journal of Alloys and Compounds* (2018), doi: 10.1016/j.jallcom.2018.03.377.

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1 **Ultrafine nanocrystalline NdFeB prepared by cryomilling with HDDR process**

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10 **Abstract:** In this work, the NdFeB ribbons ($\text{Nd}_{13.5}\text{Fe}_{73}\text{Co}_{7.5}\text{B}_6$) were first subjected to
11 the hydrogenation disproportionation (HD) to prepare disproportionation Nd-Fe-B
12 precursor and subsequently cryomilled for different times. The submicron NdFeB
13 particles with ultrafine nanocrystalline were finally obtained after desorption
14 recombination (DR) process. The results indicated that with the increase of the
15 cryomilling time, $\text{Nd}_2\text{Fe}_{14}\text{B}$ grains were refined to nanoscale, but the coercivity
16 decreased due to the strong exchange coupling between the nano-grains. The
17 optimized nanocrystalline NdFeB particles have a size of 40 nm or less and present a
18 coercivity of 6.63 kOe after cryomilling for 10 hours followed by DR treatment at
19 800°C for 10 min. To further improve the magnetic properties, Ca was added into HD
20 powders in the course of cryomilling process to protect the powders from oxidation
21 and a noticeable coercivity of 9.49 kOe was obtained. Finally, the mechanism of
22 magnetic interactions and coercivity enhancement were discussed.

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