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EFFECT OF SHOCK WAVE ACTIVATION ON NITRIDING OF $\text{Sm}_2\text{Fe}_{17}$

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Shock wave activation treatment for $\text{Sm}_2\text{Fe}_{17}$ under explosive loading (pressure 1.4~4 GPa) has been studied. After shock treatment the dislocation density in $\text{Sm}_2\text{Fe}_{17}$ was strikingly increasing, and the nitrogen absorption capacity of $\text{Sm}_2\text{Fe}_{17}$ heightens obviously no matter the nitriding condition is continuous rise temperature, isothermal or high pressure. Especially it can effectively restrain disproportionation of $\text{Sm}_2\text{Fe}_{17}\text{N}_y$. It makes Curie temperature slightly increase.

1. INTRODUCTION

Application of shock activation in materials science and industrial fabrication is very attractive research subject⁽¹⁾. A potential application of shock activation is for $\text{Sm}_2\text{Fe}_{17}\text{N}_y$. Recently, $\text{Sm}_2\text{Fe}_{17}\text{N}_y$ has been discovered to have excellent magnetic performance such as Curie temperature $T_c=470^\circ\text{C}$, a large uniaxial anisotropy field ($B_a=14\text{T}$) and a respectable spontaneous magnetization ($\mu_0 M_s=1.54\text{T}$). The theoretical upper limit on the attainable energy product is $470\text{KJ}/\text{m}^3$ (59MGOe)⁽²⁾. In addition, a substantial progress has already been reported in developing coercivity in nanocrystalline powders⁽³⁾. But for the development and application of new family of rare earth iron nitrides there are still many important subjects needed to be solved, for example, the best magnetic performance of $\text{Sm}_2\text{Fe}_{17}\text{N}_y$ can be obtained when how many does y equal to; it decomposed at elevate temperature, so it can not be sintered by normal method; the disproportionation exist during the nitriding process; a long-term stability of magnetic performance in operation needs to be determined, etc. Recent research result⁽⁴⁾ shows $\text{Sm}_2\text{Fe}_{17}\text{N}_y$ can be consolidated by explosive consolidation process. It proved that the explosive consolidation process

does not change the preformed orientation of grain, initial morphology and intrinsic magnetic properties, and the rectangle of demagnetization curve is improved to be compared with glued magnet of same powder so the maximum energy product $(BH)_{\text{max}}$ is obviously increased. Therefore, magnets of Sm-Fe-N should have the potential to compete favorably with the well-established Nd-Fe-B magnets, if a processing route can be established, which is compatible with the metallurgical and thermodynamic features of the nitride system.

In this work, we have attempted to study the effect of shock activation on nitriding of $\text{Sm}_2\text{Fe}_{17}$.

2. EXPERIMENTAL PROCEDURES

The explosive implosive shock wave was produced by slide detonation of a cylindrical charge⁽⁵⁾. The mixed explosive of RDX[#] (cyclonite) and AN[#] (ammonium nitrate fuel mixture) in different proportions were used in our experiments. The detonation velocity of the mixed explosives was measured in the experiment, and based on this the detonation parameters⁽⁶⁾ were calculated.

In order to study the microstructure a cylindrical bulk of $\text{Sm}_2\text{Fe}_{17}$ was adopted instead of powder of $\text{Sm}_2\text{Fe}_{17}$ to put into a steel capsule. The pressure distribution along the radius of cylindrical bulk was estimated to be about 1.4GPa~4GPa. Before and after explosive action some samples were taken from the bulk of $\text{Sm}_2\text{Fe}_{17}$ to be examined by micrograph, SEM, TEM and XRD. Then the bulk $\text{Sm}_2\text{Fe}_{17}$ was smashed into powder. A special phenomenon deserved extra attention, that is the smashing time of the bulk after shock action was obviously shortened by contrast to that of initial bulk. The both differ by ten times.

Nitriding experiments of $\text{Sm}_2\text{Fe}_{17}$ powder before and after shock action were respectively completed under three different conditions for the sake of contrast. They are continuous rising temperature nitriding, isothermal nitriding and high-pressure nitriding. The first two nitriding experiments were conducted by means of the thermopiezic analyser. The isothermal nitriding process at 495°C. The high pressure nitriding was in a high pressure furnace with high pure nitrogen at 0.5 MPa and 490°C to keep 3hr. The absorbing nitrogen was determined by quantitative analysis.

3. EXPERIMENTAL RESULT

Figure 1 shows nitrogen absorption characteristics, upper line for shock activation $\text{Sm}_2\text{Fe}_{17}$ powder (abbreviation: treatment powder) and lower line for un-shock-activation $\text{Sm}_2\text{Fe}_{17}$ powder (abbreviation: untreated powder). The solid line represents predetermined rising temperature line. The dotted line indicates measured rising temperature line. Table I gives experimental results about the effect of shock activation on nitriding of $\text{Sm}_2\text{Fe}_{17}$ under three different conditions. The data shows under three different condition the nitrogen absorption of shock-treatment $\text{Sm}_2\text{Fe}_{17}$ all are higher than that of untreated $\text{Sm}_2\text{Fe}_{17}$. This indicates the shock activation can increase nitrogen absorption of $\text{Sm}_2\text{Fe}_{17}$, especially under continuous rising temperature the increment of nitrogen absorption is the maximum, but the y value is not up to 3. The pressure increasing is not beneficial to nitrogen

absorption. For the sake of contrast X-ray diffraction patterns of nitriding shock-treatment powder and of nitriding untreated powder was put in one figure. Fig. 2 gives the main portion of their X-ray diffraction patterns. The contrast shows shock treatment can restrain the disproportionation of $\text{Sm}_2\text{Fe}_{17}\text{N}_y$, $\alpha\text{-Fe}$ in nitriding untreated powder is obviously higher than that in nitriding shock treatment powder under the same comparing condition, $\alpha\text{-Fe}$ exists, which shows decomposition of $\text{Sm}_2\text{Fe}_{17}\text{N}_y$ occur. This is obviously not hoped occurrence.

Fig. 3 is in illustration of microstructure change of $\text{Sm}_2\text{Fe}_{17}$ before and after shock action. TEM micrograph shows typical features of dislocation distribution found in untreated $\text{Sm}_2\text{Fe}_{17}$ (left) and in shock treatment $\text{Sm}_2\text{Fe}_{17}$ (right). It can be seen that in untreated $\text{Sm}_2\text{Fe}_{17}$ there are only a few dislocations, but in shock treatment $\text{Sm}_2\text{Fe}_{17}$ a lot of dislocations exist and cross each other. This shows shock wave action makes defect strikingly increase.

Both shock treatment $\text{Sm}_2\text{Fe}_{17}$ and untreated $\text{Sm}_2\text{Fe}_{17}$ were smashed in the same

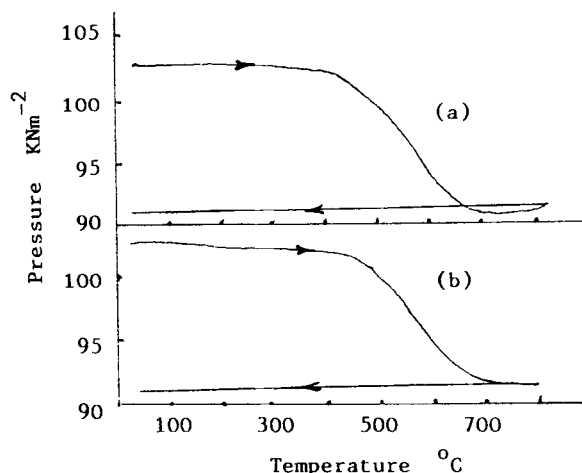


FIGURE 1. Nitrogen absorption characteristics for $\text{Sm}_2\text{Fe}_{17}$ powders heated in the thermopiezic analyser in $\sim 0.1\text{MPa}$ of N_2 (a) for shock activation $\text{Sm}_2\text{Fe}_{17}$ powder, (b) for un-shock-activation powder

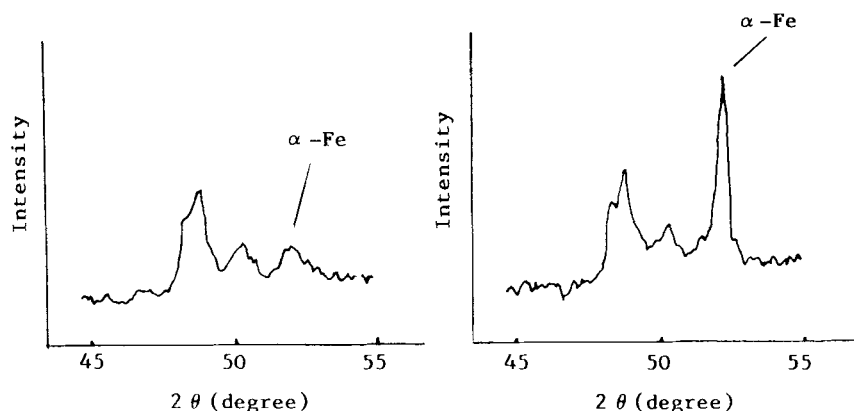


FIGURE 2. The contrast of main portion of X-ray diffraction patterns for nitriding shock treatment powder (left) and for nitriding untreated powder (right)

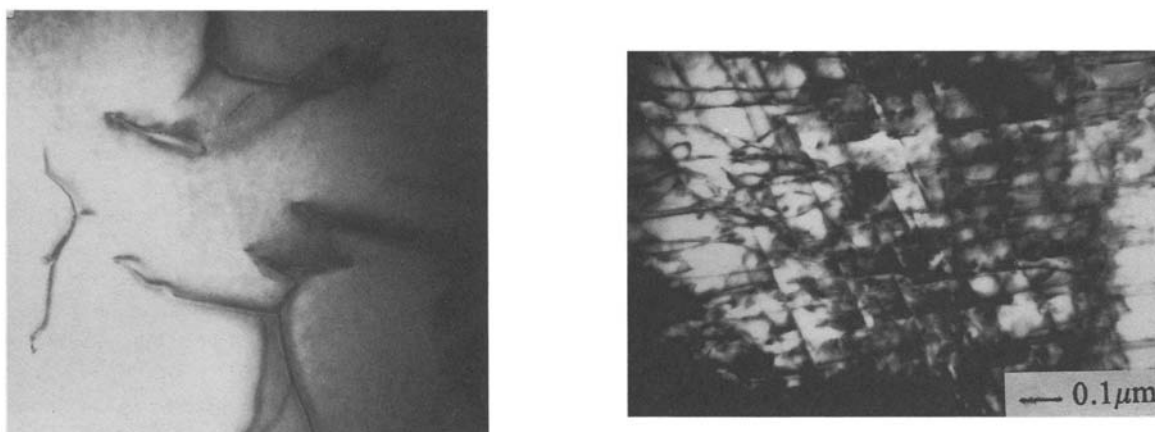


FIGURE 3. TEM bright field micrograph showing typical features of dislocation found in untreated $\text{Sm}_2\text{Fe}_{17}$ (left) and in shock treatment $\text{Sm}_2\text{Fe}_{17}$ (right)

time, so the powder size of shock treatment is finer than that of untreated. Several magnetic performance for the two powders were measured. Curie temperature of shock treatment $\text{Sm}_2\text{Fe}_{17}$ is slightly higher than that of untreated $\text{Sm}_2\text{Fe}_{17}$. Other magnetic performance do not have obvious change.

4. RESULT AND DISCUSSION

The effect of shock wave activation on nitrid-

ing of $\text{Sm}_2\text{Fe}_{17}$ is obvious. It makes nitrogen absorption increase, no matter the nitriding condition is continuous rising temperature, isothermal or high pressure. It can restrain the disproportionation of $\text{Sm}_2\text{Fe}_{17}\text{N}_y$ and increase stability of $\text{Sm}_2\text{Fe}_{17}\text{N}_y$. It makes Curie temperature slightly increase. The microstructures observed by TEM show it makes defects strikingly increase. The density and distribution of dislocation in shock treatment $\text{Sm}_2\text{Fe}_{17}$ is obviously different from that in untreated $\text{Sm}_2\text{Fe}_{17}$. The shock wave activation is evidently related to

TABLE I. Experimental results of nitriding of $\text{Sm}_2\text{Fe}_{17}$

	Continuous rising temperature nitriding	Isothermal nitriding	High pressure nitriding
Experimental condition	Continuous rising temperature ~0.1MPa of N_2	T = 768K P ~0.1MPa of N_2 3hr	P = 0.5MPa T = 763K, 3hr
Untreatment	y = 2.60	y = 2.36	y = 2.27
Shock treatment	y = 2.92	y = 2.57	y = 2.44
Nitrogen absorption increasing	12.3%	8.9%	7.5%

Note: Here y represents an amount of absorbed nitrogen atom in the structure of $\text{Sm}_2\text{Fe}_{17}\text{N}_y$ compound.

microstructure change. It is well known that the most important obstruction for application of $\text{Sm}_2\text{Fe}_{17}\text{N}_y$ is its stability (high temperature and a long period of time). The results of this study show the shock wave activation may help solve this problem.

Because smelting pure $\text{Sm}_2\text{Fe}_{17}$ is very difficult, it always accompanies with a few $\alpha\text{-Fe}$ and rich samarium phase. As relative contrast the results can still improve some problems. If mixture of NH_3 and H_2 was used instead of N_2 , a better result may be obtained.

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