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PROCESSING AND SURFACE PROPERTIES OF BASED ON IRON SINTERED ALLOYS AFTER PLASMA NITRIDING TREATMENT

PROCES WYTWARZANIA I WŁAŚCIWOŚCI POWIERZCHNIOWE AZOTOWANYCH PLAZMOWO SPIEKANYCH STOPÓW NA BAZIE ŻELAZA

Sintered parts based on iron and alloyed powders have been widely used in automotive industry. In order to enhance surface properties of those PM (Powder Metallurgy) alloys like hardness and abrasive wear, nitriding and carbonitriding treatment are applied. One of the main problem of PM chemical heat treatment alloys is their porosity degree. In the experiments Fe-Ni-Cu-Mo and Fe-Mo sintered structural parts modified by boron were made. Boron activates the sintering process which results in their considerable consolidation in the sintering at 1200 °C for 60 min. in the atmosphere of hydrogen. The experiments are related to the production of sintered structural elements based on iron powder – NC 100,24 as well as Astaloy Mo (Fe-Mo) and Distaloy SA (Fe-Ni-Cu-Mo) modified by 0.2 wt%, 0,4 wt% and 0,6 wt% B. P/M parts were obtained by mixing powders said above, followed by compacting at 600 MPa pressure and sintered at 1200 °C during 60 minutes time in hydrogen atmosphere. Selected sintered parts were plasma nitrided at 560 °C during 4 hours time. The effect of plasma nitriding on the microstructure and surface properties of samples has been analyzed.

Keywords: alloyed powders, boron, sintering, plasma nitriding, microstructure, hardness, abrasion resistance

Elementy spiekane produkowane z proszków żelaza i z proszków stopowanych znajdują szerokie zastosowanie w przemyśle motoryzacyjnym. Ażeby podwyższyć właściwości powierzchniowe stopów wytworzonych technologią metalurgii proszków, między innymi twardość i odporność na ścieranie stosuje się azotowanie lub węgloazotowanie. Podstawowym problemem obróbki cieplno-chemicznej spieków jest występowanie pewnego stopnia porowatości. W przeprowadzonych eksperymentach wytworzono spieki z proszków stopowanych (Fe-Ni-Cu-Mo) i (Fe-Mo) z dodatkiem boru, który aktywuje proces spiekania prowadzony w temperaturze 1200 °C, w czasie 60 min., w atmosferze wodoru i w efekcie doprowadza do znacznego zagęszczenia próbek. Wykonane eksperymenty dotyczyły otrzymywania spieków na bazie proszku żelaza – NC 100,24, proszku stopowanego Astaloy Mo (Fe-Mo) i proszku stopowanego Distaloy SA (Fe-Ni-Cu-Mo) z dodatkiem 0,2%wag., 0,4%wag. i 0,6%wag. Próbki uzyskano w wyniku mieszania w/w proszków z dodatkiem boru, następnie prasowania pod ciśnieniem p = 600 MPa i spiekania w temperaturze 1200 °C, w czasie 60 min., w atmosferze wodoru. Wybrane części spiekane poddano procesom azotowania plazmowego w temperaturze 560 °C, w czasie 4 godzin. W toku eksperymentów przeanalizowano wpływ azotowania plazmowego na mikrostrukturę i właściwości powierzchniowe badanych spieków.

1. Introduction

Nitriding is a process which consists in saturating the steel surface layer with nitrogen, while the object under treatment is subject to soaking for a certain time in an environment with free nitrogen atoms and other nitrogen active particles like NH^+, N_2^+. Nitrogen forms iron nitrides, which contributes to obtaining a very hard, wear resistant surface layer without any additional heat operations. The solubility of atmospheric (molecular) nitrogen in liquid iron is insignificant, and actually, of no importance. Instead, if atomic nitrogen is put into solid iron, its contents in iron may reach a level of 11.3% which corresponds to Fe_2N. The consequence of long time nitriding is the formation of a continuous zone of nitrides on steel surface, type ε ($Fe_{2-3}N$), and often – of carbonitrides $Fe_2(CN)_{1-x}$. In appropriate conditions, a film of γ' nitrides is formed under the layer of ε nitrides. This zone passes into an internal diffusion zone of ferrite supersaturated with nitrogen with precipitations of γ' nitrides and of metastable nitrides α”-$Fe_{16}N_2$. In alloy steels, in the internal zone are also present very fine

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nitrides and carbonitrided whose structure and quantity will depend on the concentration of nitrogen and alloying elements. The morphology and phase composition of the nitrided layer in steels depends upon the concentration of nitrogen in the respective zones.

Nitriding enhances the resistance to corrosion, diminishes the friction coefficient and causes a slight increase in sizes [1,2].

Various nitriding methods are applicable, viz.: gas nitriding, nitriding in powders, and glow discharge nitriding which is one of the latest nitriding technologies applicable for parts made of constructional and tool steels[3-5].

Plasma nitriding takes place in the atmosphere of ionized nitrogen in an apparatus consisting of a tight retort, vacuum pump *6*, gas feeder and proportioner *5* and electrical feeder *3* [Fig 1]. The objects subject to nitriding *2*, isolated from the retort walls are placed in the retort which also serves as an anode and is connected to the negative pole. The applied voltage is 0.5 – 1.5 kV, and the pressure of nitrogen or mixture nitrogen-hydrogen in the retort is reduced to several hPa.

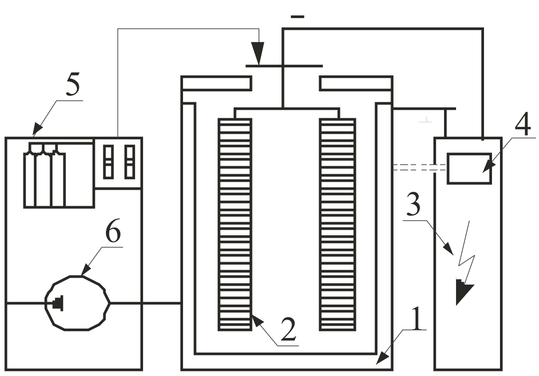


Fig. 1. Scheme of equipment for Ion-nitriding (Klockner Ionin Company) 1 – vacuum furnace with retort, 2 – charge, 3 – electrical feeder, 4 – control equipment, 5 – feeder proportioner of gas, 6 – vacuum pomp

High pressure causes ionization of gas in the narrow voltage drop zone at the cathode. Collisions of nitrogen ions with the surface of the object under treatment – occurring on the cathode – lead to the release of heat, which will heat up the object under treatment to the nitriding temperature.

Iron atoms, beaten out of the surface, form nitrides which are then subject to condensation on the metal surface with subsequent decomposition by making use of atomic nitrogen that diffuses inside the metal. Those phenomena may be controlled by changing the voltage, pressure and composition of the gas, which enables one to control the surface layer structure and to produce – on the surface – either a continuous zone of ε and γ’ nitrides or only a diffusion zone [6]. Surface layers produced in that process are characterized with high wear resistance, fatigue strength and appreciably higher plasticity if compared with layers obtained if nitriding processes are performed with other methods [7-8].

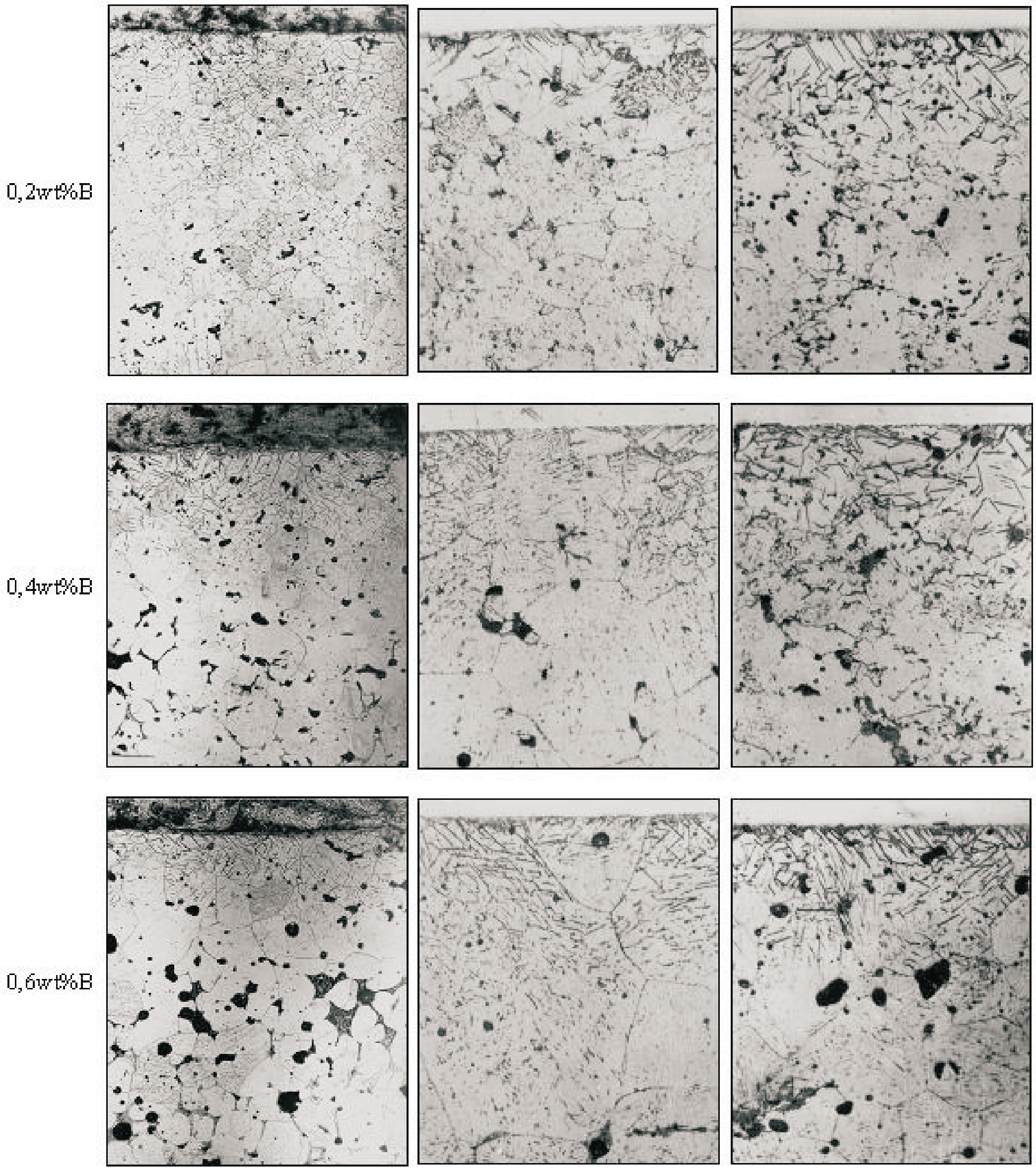
A large number of automobile products are made from iron-based alloying powders; that is why in recent years there have been performed experiments aimed at using plasma nitriding processes for metal powder sinters [9-13]. Recently, in many cases Ti alloys are covered by thin layers via glow discharge and laser treatment [14-15]. This paper deals with plasma nitriding processes of sinters made from iron powders and boron-modified alloying powders. An analysis was also made in order to determine the effect of heat and chemical treatment upon microstructure and surface properties of sinters under investigation.

2. Experimental procedure and results

The following powders were subject to research: iron powder NC 100.24 (Hoeganas), alloyed powders: Astaloy Mo (Fe-1.5% Mo) and Distaloy SA (Fe-1.75 %Ni-1.5%Cu-0.5%Mo) – (Hoeganas) and boron powders (CERAC). Sintered parts were obtained by mixing powders of iron and alloy powders with some boron addition (respectively 0.2, 0.4 and 0.6%) in a Turbula mixer; the operation of compaction was effected on a hydraulic press at p =600 MPa in order to produce cylindrical samples. The sintering process took place at 1200°C in hydrogen atmosphere for a 60 minutes’ time. The final sinters were subject to heat and chemical treatment, viz. plasma nitriding at 560°C (4 hours’ time). Experiments allowed to make the structural investigations and the density, microhardness and wear resistance properties of samples. Structural investigations of samples were made on light microscope type Neophot 32, the density of sinters was determined by means of Archimedes’ method in conformity with PN-EN ISO 2738, December 2001, microhardness of samples – immediately after sintering, and then after nitriding (Vickers’ method) on a ZWICK hardness tester coupled with a PC. Instead, for tribological tests (measurement of friction coefficient was used a four ball apparatus manufactured by ITE (Institute of Technology and Operation] Radom (applied were bearing balls, dia. $^1/_2$”, made from steel ŁH15).

A high compaction degree is related to the sintering process of samples under investigation. While sintering materials made from the following powders: iron NC 100.24, Astaloy Mo (Fe-Mo) and Distaloy SA (Fe-Ni-Mo-Cu) with boron addition at 1200°C, an eutectic reaction between iron and Fe_2B leads to the formation of a liquid phase which causes an appreciable compaction, and at the same time, a decrease in porosity.

The said process is very significant in the case of nitriding sintered materials [7,8]. While nitriding sinters, an important role is played by porosity. It should be tried to obtain the lowest porosity possible so that nitrogen may diffuse into the surface under treatment as well as possible. It is exactly the process of sintering of structural parts with boron additions to produce an appreciable compactness and reduction of the open porosity [Fig. 2]. The morphology of the porosity changed from open to closed, and round upon the additions of boron however, liquid phase sintering in the presence of the highest boron contents, enables dramatic grain growth, especially in the Astaloy Mo samples in which a reduced amount of alloying elements (if compared with Distaloy SA samples) did not obstruct this growth [12]. The phase analysis showed that a diffusion layer was composed of an ε solid solution and Fe_4N (γ') nitride, also probably boron nitride (BN) [9, 11,12]. Presence of BN in the structure of tested plasma nitrided sinters influence on the increasing of their microhardness and friction coefficients.



a)
(NC 100,24)

b)
(Astaloy Mo)

c)
(Distaloy SA)

Fig. 2. Microstructures of sinters with 0,2 wt%, 0,4 wt% and 0,6 wt% of boron after plasma nitriding: a) Fe-NC 100,24, b) Astaloy Mo, c) Distaloy SA

With increasing the boron contents from 0 to 0.6%, the samples of Fe-NC 100.24 , Astaloy Mo and Distaloy SA were found to have an appreciably higher microhardness value in sinters both before and after nitriding. Be also stressed that higher microhardness values were obtained after a heat and chemical treatment [Fig 3-5].

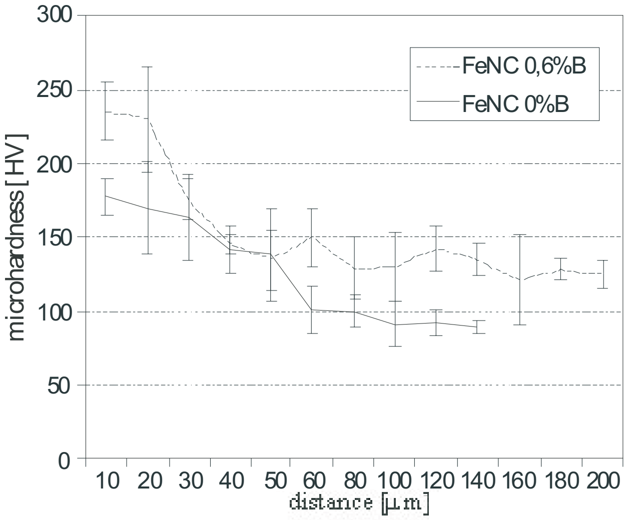


Fig. 3. Microhardness profile of Fe-NC 100,24 sinters with 0 wt% and 0,6 wt% of boron after plasma nitriding

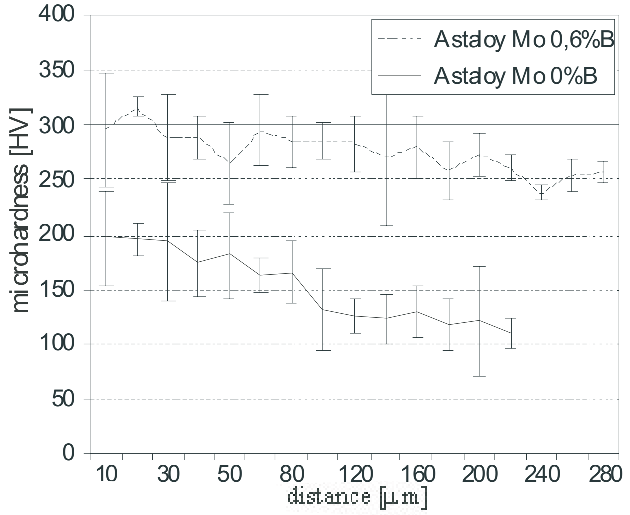


Fig. 4. Microhardness profile of Astaloy Mo sinters with 0 wt% and 0,6 wt% of boron after plasma nitriding

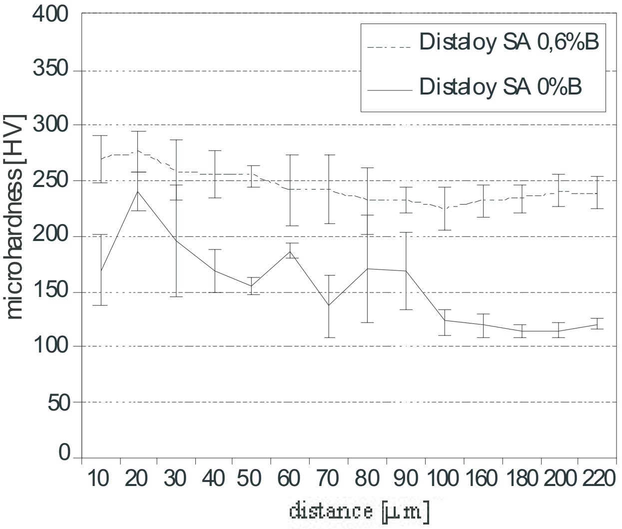


Fig. 5. Microhardness profile of Distaloy SA sinters with 0 wt% and 0,6 wt% of boron after plasma nitriding

Averaged results for friction coefficient for sinters of Fe-NC 100.24, Astaloy Mo and Distaloy SA with input boron level of 0.6% and after plasma nitriding (0 and 0.6% B) are illustrated in Figures 6-8. The graphs were obtained according to 15 averaged measuring points (a measuring series consisted of 450 points).

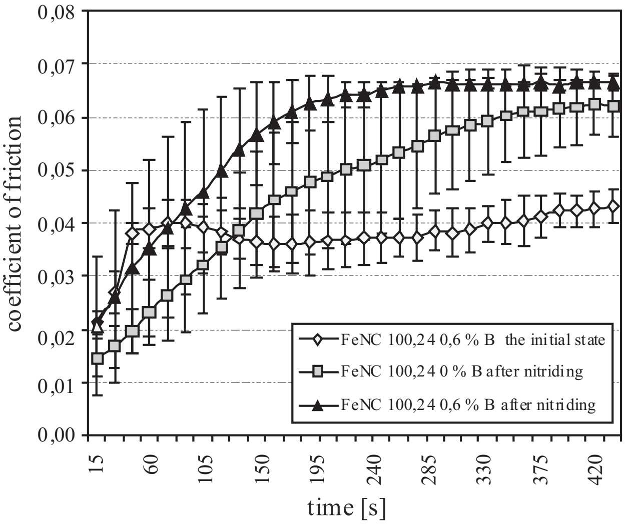


Fig. 6. Dependence between coefficient of friction and a time for Fe – NC 100,24 with 0,6 wt% of boron after sintering and with 0 wt% and 0,6 wt % of boron after plasma nitriding (dependence diagram was established base on 15 averaging of measurement points)

Tribological tests performed on sinters revealed an increase in friction coefficients for all nitrided samples, and the highest increase was observed for the sinters made from Fe-NC 100.24 powder [Fig. 6], followed by Distaloy SA [Fig. 7] and Astaloy Mo [Fig 8]. There was observed an increase in friction coefficients for all samples under investigation if subject to nitriding. It was also found that as boron contents rose in the nitrided sinters under analysis, the friction coefficient also increased. The said relation is also connected with the porosity of sinters. And namely, for lower porosities the friction coefficient was higher. Therefore, the friction coefficient in nitrided sinters depends on the boron contents and on the degree of their porosity.

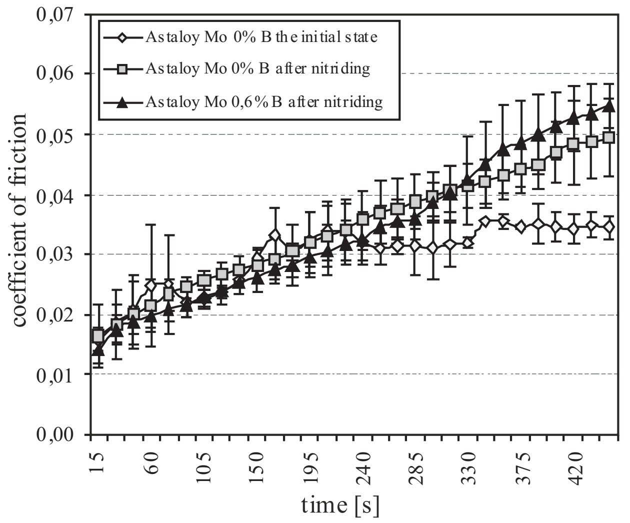


Fig. 7. Dependence between coefficient of friction and a time for Astaloy Mo with 0,6 wt% of boron after sintering and with 0 wt% and 0,6 wt % of boron after plasma nitriding (dependence diagram was established base on 15 averaging of measurement points)

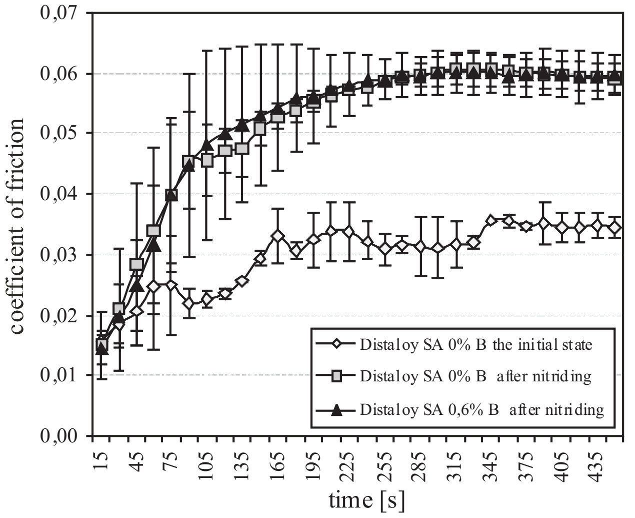


Fig. 8. Dependence between coefficient of friction and a time for Distaloy SA with 0,6 wt% of boron after sintering and with 0 wt% and 0,6 wt % of boron after plasma nitriding (dependence diagram was established base on 15 averaging of measurement points)

The fluid used for tribological tests is likely to have penetrated into pores in the case of sinters with higher porosity level, and to have thus decreased the friction coefficient. In turn, in nitrided sinters with lower porosity degree, sinter grains might have not only crumbled acting as extra abradant, but also increased the friction coefficient.

The mentioned effect depends as well upon the composition of input powders and phase composition of sinters. That is why for the successive research stage are scheduled investigations into the structure and phase composition of sinters before and after nitriding as well as analyses of the influence exerted by the structure and phase composition of sinters upon their surface properties (hardness and wear resistance).

3. Summary

In the process of plasma nitriding of sinters made from the following powders: Fe-NC 100.24 , Astaloy Mo and Distaloy SA porosity is deemed to play an important role. In the sinters with the lowest degree of porosity, nitrogen had diffused correctly into the surface under investigation. The sintering process performed in products made from powders Fe-NC 100.24, Astaloy Mo and Distaloy SA with boron addition causes an appreciable compaction and reduction of open porosity. The sinters under investigation subject to plasma nitriding proved to have enhanced microhardness values; but higher microhardness values were obtained for sinters with higher boron contents. Tribological tests carried out on nitrided sinters indicate an increase in the friction coefficient, which is quite surprising. The said phenomenon may arise from the fact that input samples after sintering were characterized with a certain degree of open porosity. The fluid used for tribological tests is likely to have penetrated into pores in the case of sinters with higher porosity level, and to have thus decreased the friction coefficient. In turn, in nitrided sinters of lower porosity degree, sinter grains might have crumbled acting as extra abradant, and in consequence, could have increased the friction coefficient. The effect mentioned depends as well upon the composition of input powders and phase composition of sinters (ε) solid solution, Fe_4N (γ') nitride and probably boron nitride (BN).

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