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1. Development and characterization of cubic boron nitride based surface composite on D2 tool steel using thermal diffusion, https://sciedirect.com/science/article/pii/S2214785320312086

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Synthesis of cubic boron nitride diffused-D2 steel surface composite by thermo-chemical diffusion process to enhance the wear resistance

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Abstract
The diffusion of cubic boron nitride (c-BN) on the D2 tool steel surface was investigated using a thermo-chemical process in order to enhance the tribological properties of tool steel surfaces. The c-BN was diffused on the tool steel surface applying the thermochemical diffusion process using argon controlled furnace. The effect of temperature and soaking time on the diffusion process, micro-hardness, and wear resistance were the main parameters evaluated. The field-emission scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) was used to analyze the morphology and elemental composition of the as-synthesized c-BN-D2 surface composite. Here, the micro-hardness of manufactured coatings was investigated using a Vickers hardness tester. The wear resistance of surface composite was studied using a pin-on-disk apparatus. The morphological investigation revealed that c-BN was successfully diffused in the surface of tool steel in the form of white globular precipitates. The best surface hardness produced has a values of 1570 GPa. The diffused layer thickness varies between 50–90 μm and is very dense. It has mechanical interlocking properties suitable against delamination process and wear formation. The superior performances of novel c-BN-D2 tool steel surface composite permits to extend the machine tool life, especially when is applied on the single-point cutting tools.

1. Introduction
High temperature machine tools are manufactured from tool steels. Typically, these tools are identified in components such as single-point cutting tool, drill bit, and milling cutter, etc [1]. Despite their potential in numerous applications, the low surface hardness (330–330 HV) of tool steels cause massive limitation to its large industrial use [2]. Several efforts have been reported to improve the surface properties of tool steels by applying heat treatment [3], coating [4], and cladding [5]. The heat treatment is recognized in normalizing, annealing, hardening, tempering, stress-relieving, and case hardening treatments. It helps to enhance the surface characteristics; however, they may have limitation because (i) time-consuming process, (ii) metal may get oxidized, (iii) or some of the material properties are lost [6–8]. The major coatings used are plasma spray deposition, chemical vapor deposition (CVD), physical vapor deposition (PVD), and electro-deposition process [9, 10]. The main coatings were achieved using metallic and ceramic materials such as CrN, Al2O3, WC, TiAN, and TiC, etc. By applying these coatings, the wear resistance and friction can be improved slightly. Yet, the coatings may have some disadvantages such as high porosity generation, low density, formation of high amount of oxides, lower bond strength, hard to control coating thickness and uniformity [11, 12]. The coating thickness
and consistency may be managed at the expenses of increasing the prices of procedures and/or losing some mechanical surface properties [13–15].

Recently, some improvement on the surface properties were achieved through novel innovative manufacturing processes like powder metallurgy, friction stir processing, and diffusion through the furnace. Among these routines, the diffusion technique is considered the better one which is also a cost-effective procedure [16, 17]. This thermal diffusion technique is an advanced surface-modification process. It improves the surface properties by developing surface-composite which provides safety against harsh environmental conditions for industrial components [18, 19]. Different compounds formed using this technique (i.e. thin layers of carbides, borides, aluminids, and chromides) allows better mechanical, corrosion, wear resistance, and tribological properties of steel and other metal substrates [20–24].

The thermochemical diffusion technique is a concept derived from CVD process. It involves a chemical alteration of steel substrates when is exposed at elevated temperatures (e.g. 800 °C–1100 °C). During this process, the foreign particles placed on the surface get diffused in the newly surface [19, 29–30]. Galedar et al [31] reported a critical review on the diffusion of boron and intermetallics compounds (i.e. aluminids and chromides) on the steel surface. In their review were presented the main potential of these diffused compounds. Moreover, cubic boron nitride (c-BN) is considered a viable alternative as coating and reinforcement material in order to enhance the mechanical properties of tool steel used for cutting tool applications [32, 33]. Up to now, only several techniques were used for deposition of c-BN to solve various industrial needs [34–37]. Saggau et al [38] achieved a surface composite by diffusing c-BN on D2-tool steel using a furnace. Their results are promising cause the c-BNs were successfully distributed to the D2-steel surface.

To contribute to further development and application of c-BN thermo-chemical diffusion process at an industrial scale, in this work a detailed research on the mechanical and tribological properties of c-BN-D2 surface composite were proposed. The mechanical characteristics were evaluated through hardness test and tribo-wear tests. The wear and friction mechanism were explained using optical observation. The results endorse the c-BN thermo-chemical diffusion technique as a viable alternative to create superior wear resistance components produced at cost effective.

2. Materials and Method

In this study, D2 tool steel was used as a substrate surface. The samples of 20 mm diameter and 15 mm thickness were cut by using a wire-EDM process. Figure 1 shows the flowchart of methodology adopted for c-BN layer diffusion and analysis. As preliminary step of diffusion technique, the surface of D2 tool steel samples was grounded to achieve plane surface and a surface roughness \( R_a \) of 1.5 \( \mu m \). Figures 2(a), 2(b) presents the geometry, size, and structure of c-BN powder particles. The c-BN compounds were analyzed by x-ray Diffraction (XRD) and were included in figure 2(c).

In order to create the surface composite a controlled vacuum furnace were used in which were placed the D2-tool steel samples. During the thermo-mechanical process the vacuum furnace was pre-heated and the c-BN powder particles were sprinkled on the surface of D2-steel samples. The effect of temperature (550 °C–950 °C) and holding/soaking time (0.5 to 5 h) were analyzed. The diffused samples were then cooled for 18 h in the furnace. Figure 3 shows the main experimental set-up of thermo-chemical diffusion process and the schematic representation of the diffusion route via a vacuum furnace. In table 1 were presented the thermal diffusion process parameters and the out-put response characteristics.

The surface morphology and elemental composition of the as-prepared surface composite were investigated using field-emission scanning electron microscopy (FE-SEM; JEOL 7600F) which is coupled to energy dispersive spectroscopy (EDAX). The thickness of the diffused layer was measured by taking the cross-section of the coated samples. There, the coated samples were cut using a low-speed diamond cutter and subsequently ground using 1/0, 2/0, 3/0, 4/0, and 5/0 graded emery paper. Finally, the polishing of grounded samples was carried out using the diamond paste in order to obtain a mirror-like super finished surface [39]. Then, the polished cross-section of the coated surface was investigated under FE-SEM to examine the morphology of diffused layer thickness.

Micro-hardness was determined under a Vicker micro-hardness tester using a 0.200 gm load, which was hold for 15 s. The micro-hardness was evaluated on the cross-section of the c-BN diffused layer. A well-grounded and polished cross-section were prepared for indentation procedure. The wear test was conducted on a pin-on-disc wear test rig (DUCOM, Instruments Pvt. Ltd, Bangalore, India) [20, 33] to evaluate the as-prepared surface composite for tribological characteristics. In the wear trials was used a pin of \( \phi 5 \times 10 \) mm prepared through diffused technique. The pin was rubbed against WC, \( Al_2O_3 \), \( Si_3N_4 \) counter surface material for a sliding distance of 500 m. The sliding velocity imposed during the wear test was 0.1 m s \(^{-1}\). The amount of specific wear rate, \( K' \) (\( mm^3 \times N^{-1} \times m^{-1} \)) and the coefficient of friction (COF) was determined accordingly.
3. Results and discussions

Table 1 shows the effect of temperature and soaking time on diffusion of c-BN applied on the D2-tool steel surface. There was no visual diffusion observed at 550 °C and 650 °C for different soaking time. We speculate that this is because the recrystallization temperature of steel is higher than 650 °C, which may trigger difficulties in occurring the thermodynamically process. Thereby, the c-BN was unable to dissolve on the surface. We have noted that the diffusion of c-BN is produced at or temperature higher than 750 °C for soaking of at least 2 h. At 750 °C, the D2 steel undergone through the re-crystallization phase. It is slightly softer cause the c-BN particles reinforced into the substrate surface are up to few microns. We have noted that the diffusion performance depends of temperature and soaking time. Longer soaking time (e.g. more than 2 h) may generate a significant microstructure transformation and a thicker diffusion layer. Here, the best condition for the diffusion of c-BN on the D2-steel surface was found applying the treatment at 750 °C for 2 h or 850 °C for 1 h. Figure 4 shows the optical micrograph of surface composite subjected to different temperature and soaking time during thermo-chemical diffusion treatment. It reveals details of the microstructure formation and diffusion layer patterns. The optical micrograph (figures 4(a) and (d)) from surfaces treated at 750 °C or 850 °C for a soaking time of ½ h shows no diffusion of c-BN particles on the surface. By using 750 °C and extending the soaking time to 1 h were observed the occurrence of some preliminary particle diffusion (see figure 4(b)). By setting the temperature of 750 °C for a soaking time of 2 h were possible to obtain an uniform c-BN diffused surface (figure 4(c)). Further, by increasing the temperature to 850 °C was possible to achieve an uniform c-BN diffused surface only using a soaking time of 1 h. But, a detrimental growth of c-BN particles on the surface were detected by setting the
temperature to 850 °C for 2 h of soaking. The bigger size of particle on the surface can induce a brittle behavior. At lower temperature the diffusion of c-BN occurs very slow which require longer soaking time (i.e. 2 h). However, a higher temperature facilitates fast diffusion of particles and the complete diffusion happen by using one hour of soaking time. Th longer soaking time (2 h) at higher temperature enables the occurrence of bigger particles on the surface which can deteriorate the mechanical properties of treated material.

Figure 5 shows the microstructure formed on the cross-section of the diffused layer and substrate surface. These SEM micrographs enable to identify the morphology of diffused c-BN on the D2-steel surface. The diffused c-BN appears as bright grey color while the substrate surface is developed as dark structure. There, the yellow color arrows highlight the diffusion of c-BN. By comparing the microstructure from figures 5(a) and (b) we can indicate that the distribution of c-BN is higher on the samples treated at 850 °C for 1 h. Overall, the microstructure examined is highly dense on the entire surface. During the thermo-mechanical process the solidification occur, and the re-crystallization allows to merge the grain between D2 steel and c-BN particles leading to c-BN diffusion on the D2-steel matrix. Figures 5(c) and (d) show the cross-section micrograph of the diffused layer. These samples micrograph allow to observe details of diffusion zone, thickness of diffusion zone and how the interface develop during the thermo-mechanical process. Figure 5(c) presents the as-treated surface composite subjected to 750 °C for 2 h which contain unevenly distributed micro-cracks and micro-holes of different sizes. The measurements prove that the c-BN particles were diffused in D2 steel substrate up to 50 μm depth. Instead when was analyzed figure 5(d) which provides details of treated surface to 850 °C for 1 h a much thicker layer of c-BN particles were noted. This later layer formed from c-BN particles diffused on D2 steel substrate reach a depth of 90 μm that is 80% higher compared to surface composite subjected to 750 °C for 2 h.
We speculate that at higher temperatures, the D2 steel material structure is susceptible to much recrystallization process. It can lead to significant c-BN particle diffusion which finally enable forming a thicker layer.

Figures 6(a)–(c) and 6(b)–(d) show the EDS analysis of the diffused layer of c-BN formed at 750 °C for 2 h and 850 °C for 1 h, respectively. The EDS analysis obtained from the substrate indicated as major substrate elements (Fe, C). On the other hand, the EDS data obtained from the layer formed of c-BN particles indicated the increase of elements such as boron and nitride, which confirms that only high purity c-BN were reinforced. By evaluating the elements quantitively from the surface treated at 750 °C for 2 h it was detected the following elemental wt. % of C, B, N, and Fe which are 14.57, 29.57, 10.21, and 45.65, respectively. A slightly increase on the intensity of c-BN diffusion were achieved at higher temperature. Therefore, the surface treated at 850 °C for 1 h indicated the following elemental wt. % of C, B, N, and Fe which are 18.15, 35.65, 14.54, and 31.66, respectively.

Figure 7 shows the variation of surface micro-hardness across the diffused c-BN layer. The obtained results indicated that the hardness of composite samples was improved considerably as compared to raw samples. The average of five readings was taken and the mean hardness was determined. The c-BN-reinforced layer formed at 850 °C for 1 h was 1570 HV, which is 444.82% higher as compared to raw samples hardness of 303 HV. However, the measurements form the samples treated at 850 °C for 2 h reveal a slightly decrease on the hardness which probably is caused by the mismatch between D2 structure and c-BN particles formed in large sizes (see details in figure 4(f)). The mean hardness of c-BN-reinforced surface composite formed at 750 °C for 2 h was measured around 1350 HV, which is 345.54% higher as compared to the untreated samples (303 HV). This is
Table 1. Effect of temperature and soaking time on the diffusion of c-BN.

| Soaking time (hours) | Diffusion temperature (°C) |
|----------------------|-----------------------------|
|                      | 550 °C | 650 °C | 750 °C | 850 °C |
| ½ h                  | No visual change/diffusion | No visual change/diffusion | No visual change/diffusion | No visual change/diffusion |
| 1 h                  | No visual change/diffusion | No visual change/diffusion | No visual change/diffusion | Diffusion occurred |
| 2 h                  | No visual change/diffusion | No visual change/diffusion | Diffusion occurred | Diffusion occurred/change in dimensions |
| 3 h                  | No visual change/diffusion | No visual change/diffusion | Diffusion occurred/bigger c-BN particles | Diffusion occurred/bigger c-BN particles |
| 5 h                  | No visual change/diffusion | No visual change/diffusion | Diffusion occurred/bigger c-BN particles | Diffusion occurred/bigger c-BN particles |
Figure 4. Optical image of diffused c-BN treated at 750 °C and 850 °C for different soaking time: (a), (d) for ½ h, (b), (e) for 1 h and (c), (f) for 2 h, respectively.

Figure 5. SEM micrograph, and cross-section of diffused layer treated at (a), (c) 750 °C for 2 h, and treated at (b), (d) 850 °C for 1 h, respectively.
because the novel formed surface contains uniformly dispersed c-BN hard particles (see details in figure 4(f)), which helps to enhance the surface properties of D2 steel surface. Nevertheless, a longer soaking time (3 h) for c-BN treated at 750 °C seems detrimental for mechanical strength of diffused zone. The weaker properties may be explained because the formed surface at longer soaking time may contains bigger c-BN hard particles (as per table 1 details) with inferior bonding performances with the matrix material, un-melted particles and structural porosity which in turn leads to lower surface hardness.

Figure 8 shows the wear rate and average coefficient of friction of un-treated surface and c-BN diffused samples treated at 750 °C for 2 h, and 850 °C for 1 h against WC, Si₃N₄, and Al₂O₃ counter surface, respectively. The hardness measurements of WC, Si₃N₄, and Al₂O₃ counter-surface indicated a hardness in the range of 2700 HV, 1600–2000 HV, and 1700–2200 HV, respectively. Figure 8(a) indicate the wear rate for all the samples. There, we can note that the higher wear was achieved against the WC counter surface while the Si₃N₄ counter surface have the best wear resistance. This is because Si₃N₄ provides superior lubrication mechanism which leads to longer surface resistance [37]. Yet, the untreated samples have the highest wear rate in comparison to c-BN diffused samples. Obviously, the untreated samples have lower surface hardness which deteriorates faster at the contact with harder surface of counter surfaces. Here, it was found that the untreated samples have the highest wear rate against the WC counter surface. The samples treated at 850 °C for 1 h revealed the lowest wear rate which is associated to higher surface hardness as was found in figure 6(a). Generally, the c-BN may have a better surface resistance to abrasion [37]. Figure 8(b) shows the variation of coefficient of friction (COF) of untreated surface samples, c-BN diffused samples at 750 °C for 2 h and 850 °C for 1 h against WC, Si₃N₄, and Al₂O₃ counter surface. It can be seen that the untreated samples have the highest COF against the Si₃N₄ counter surface and WC counter surface. The c-BN diffused samples showed far better COF which is almost half when comparing to untreated samples. The highest COF were detected for c-BN diffused samples against Si₃N₄ counter surface as compared to WC and Al₂O₃. The variation of coefficient of friction is attributed to fracturing and removal of c-BN particles from the surface which form the third body effect. Thus, they get detached against
Figure 7. (a) Micro-hardness versus different depth of diffusion from the top surface to substrate, (b) untreated surface, (c) samples treated at 750 °C for 2 h, and (d) samples treated at 850 °C for 1 h.

Figure 8. (a) Wear rate and (b) average COF of untreated surface, c-BN diffused samples at 750 °C for 2 h, and 850 °C for 1 h used against WC, Si₃N₄, and Al₂O₃ counter surface.
high strength surface (Si$_3$N$_4$) generating higher friction values. A similar observation was found in the literature [20, 36].

Figures 9(a) and (b) show the SEM morphology of the worn surface of counter plates WC and untreated pin samples. Figure 9(a) micrograph reveals almost no worn-out effect on the WC-counter surface. Instead, some material deposition from the untreated pin was observed. This is because the untreated pin has a lower surface hardness and obviously may fail at the contact with the WC-counter tool. Additionally, it causes ploughing and robbing for untreated pin surface as depicted in figure 9(b). The wear track evaluated from the WC-counter surface used against the samples treated at 750 °C for 2 h reveals the rubbing marks and deposition of c-BN debris (see details in figure 9(c)). There, during the tribological contact some c-BN particles start to fracture early
because the hardness of c-BN diffused layer (1350 HV) is much lower when comparing to WC (2700 HV). Thereby resulting in the occurrence of third body that gradually start to stick on the counter surface materials (our case WC). The investigation of pin treated at 750 °C for 2 h subjected against WC confirms the material removal and also shows the occurrence of craters and scratch marks (see details in figure 9(d)). Additionally, on the pin surface were noted also micro-size pits and sub-micron size debris. They are identified especially on the location where the c-BN particles were removed. We speculate that the sub-micron size debris may come from the WC oxide which get fractured during the tribological process. Even so, the measurements indicated that the wear resistance is improved with almost 82.75% by diffusion of c-BN in D2 steel, for the pin samples treated at 750 °C for 2 h, when comparing to untreated pin samples. Instead, the wear track evaluated from the WC-counter surface used against the samples treated at 850 °C for 1 h reveals slightly more accentuated rubbing marks and less deposition of c-BN particles (see details on figure 9(e)). This is probably because these samples possess a better interface bonding between the c-BN and matrix which results in a denser structure (as shown in figure 3(e)). Therefore, a smaller number of c-BN particles may get removed from the treated surface. The evaluation of pin samples surface treated at 850 °C for 1 h shows comparatively fewer craters and scratch marks (see details on figure 9(f)). The quantitative measurements indicated that the wear resistance is improved with almost 89.65% by diffusion of c-BN in D2 steel, for the pin samples treated at 850 °C for 1 h, when comparing to untreated pin samples. The results confirm that the samples manufactured at 850 °C for 1 h are better in terms of wear resistance when comparing with the samples manufactured at 750 °C for 2 h. The as-proposed technology successfully allows to create industrially c-BN diffused D2 steel tools used in high-temperature machining applications.

4. Conclusions

In this work, a surface composite was successfully developed by diffusing c-BN particles in the D2 steel surface. This was obtained by thermal diffusion process in the vacuum furnace. The main outcomes achieved from this study are presented as follows:

1. The best condition for the diffusion of c-BN in the D2 steel surface was 750 °C for 2 h and 850 °C for 1 h. Throughout, the c-BN particles were successfully diffused in the surface of steel.
2. The produced diffused layer contains uniformly distributed c-BN particles and is very dense. The diffusion zones layer was measured in the range of 50 to 90 μm, which possessed excellent mechanical properties.
3. The average hardness of the samples manufactured at 750 °C for 2 h and 850 °C for 1 h was measured around 1350 HV and 1570 HV, respectively. The improvement in the hardness for these samples was 345.44% and 444.82%, respectively, as compared to untreated samples 303 HV.
4. The novel manufactured surface composite showed excellent wear resistance characteristic. These samples revealed slightly lower wear resistance against WC, Al₂O₃ counter surfaces and the best wear rate against Si₃N₄ counter surface. Overall, the wear resistance of pin surface treated at 850 °C for 1 h was improved with almost 89.65% by diffusion of c-BN in D2 steel, when comparing to untreated pin samples.

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