Characterization of Sintered Titanium-Nickel-Diamond Grinding Layers

B. Denkena, A. Krödel, F. L. Kempf*

Leibniz Universität Hannover, Institute of Production Engineering and Machine Tools, Hannover, 30823 Garbsen Germany.

* Corresponding author. E-mail: kempf@ifw.uni-hannover.de Manuscript submitted December 15, 2021; accepted May 8, 2022. doi: 10.17706/ijmse.2022.10.2.18-32.

Abstract: This paper explores the influence of the manufacturing parameters and the bond composition of diamond grinding layers on the bond cohesion and strength. A focus is set on the investigation of carbide formation in the interface between bond and diamond grains, which is enabled by active bond materials. The use of active bond materials such as titanium increases the amount of stress the grinding layer can withstand above the point that can be explained solely by the bond strength itself. X-ray diffraction measurements were able to show the formation of titanium carbide in individual cases. The mechanical characterization via three-point bending tests, however, indicated additional effects on the overall cohesion inside the grinding layer, besides the impact of the titanium content alone. Scanning electron microscope micrographs also revealed increased breaking of diamond grains under mechanical load in nickel-titanium bonds compared to bronze bond, which can indicate a stronger adhesion between bond and grain.

Key words: Diamond composites, grinding tools, sintering, characterization.

1. Introduction

Unless fillers are added in the manufacturing process, grinding tools with sintered metal bonds consist of a dense metallic bond matrix that shows little porosity, especially when compared to most vitrified bonds [1]. This is linked to the fact that metallic grinding wheels are commonly manufactured by hot pressing, which actively compresses the sintering mold, improving the contact between the metal particles [2]. This ultimately results in a low porosity of the grinding layer. The metallic bond system is commonly applied in combination with superabrasive grains from diamond or cubic boron nitride. The combination of diamond grains with certain elements can introduce chemical side reactions into the otherwise rather simple thermomechanical process, making it harder to predict the resulting grinding layer.

The sintering process uses elevated temperatures, which can pose a problem when the metastable diamond is used as an abrasive. According to literature, the phase transition into the thermodynamically stable carbon modification of graphite takes place above approximately 973 K (700 °C). In the case of nanodiamonds, this transition can occur at lower temperatures of 873 K (600 °C) [3] depending on the surrounding conditions. Besides the temperature, some elements promote the phase transition of diamond to graphite. An investigation of several elements that are commonly used in binder materials was used to determine the carbon reactivity under sintering conditions. It concluded that under similar conditions cobalt, iron, and nickel lead to the formation of graphite, whereas copper and chromium did not. This is due to the different catalytic activities. Moreover, the formation of carbides of chromium under sintering conditions was shown [4].

Carbon can form a wide variety of carbides with different elements. These can be subdivided into different classes based on their general properties. For this paper, the major classes are covalent, salt-like, and interstitial (sometimes: metal-like) carbides. Typical examples for covalent carbides are silicon carbide (SiC) and boron carbide (B12C3). These are stable in the presence of water and show a high hardness. Therefore, they are commonly used as abrasives. Salt-like carbides are more brittle and react with water forming carbohydrates. Aluminium carbide (Al4C3) for example forms methane in the presence of water, whereas calcium carbide (CaC2) is used to generate ethyne (acetylene). Interstitial carbides like tungsten carbide (WC) and titanium carbide (TiC) have metallic properties. They exhibit high hardness, have high melting points and are considered refractory [5]. These interstitial carbides are formed by transition metals and show very different chemical behavior regarding their formation. The direct comparison between different transition metal carbides shows very large differences between the respective Gibbs energies of their formation. Ellingham diagrams show generally low values (the equilibrium of the reaction lies on the side of the carbide) for carbides like TiC, V2C and Ta2C, while other carbides like Co3C, Ni3C show positive values, indicating their thermodynamic instability. [6] These values vary for each carbide depending on the process temperature.

The reaction between diamond and carbide forming elements has already been investigated for some applications. Grinding wheel bond compositions with a low amount of cobalt, nickel, and titanium were investigated to develop enhanced bond properties [7]. The degree of wear resistance of the manufactured bond-diamond composites was tested via abrasive blasting, during which it exhibited a differing increase in erosion rate compared to a pure cobalt bond. For this purpose titanium hydride was used to produce an active solder which fixes the diamond grains to the cutting wire itself [8]. In addition, the stable carbon modification graphite reacts with titanium to form carbides. It could be shown that graphite forms carbides with titanium both, induced by temperature (600 – 1,470 K) [9], and induced by ion beam irradiation at room temperature [10]. The thermal conductivity of materials containing diamond and a metal matrix is limited by the interface between both. The in situ formation of carbides in the interface leads to improved thermal conductivity [11]. In general, these concepts are not limited to titanium. For sintered copper-diamond composites, the addition of elemental boron [12] or chromium [13] also increases the thermal conductivity and the bending strength. However, thermal damage and the graphitization of diamonds are pronounced in the sintered composites since the reactions require elevated temperatures (>973 K) to take place at a reasonable rate. Besides the aforementioned influences of composition and sintering temperature - and ultimately sintering time - more aspects can impact the degree of damage to the diamond. An improvement of the spark plasma sintering process (SPS) called spark plasma consolidation process (SPC) can further minimize graphitization [14]. This work also shows the protecting capability of titanium-coated diamond grains. Diamonds with coatings of titanium or titanium carbide are commercially available. They are marketed as resilient against corrosion of the grain, especially in the presence of iron, chromium, or Tungsten at high temperatures. Experiments concluded, that Ti- and TiC-coatings are suitable for the protection of diamond grains against the corrosion by iron in a hot pressing process. However, Ti-coated diamond grains demonstrated better performance concerning rupture stress, yield strength, and abrasion resistance [15].

Mechanical characterization of sintered bronze bonded grinding layer samples and the grinding application of the corresponding grinding wheels was able to show a correlation between critical bond stress and the dressing result [16]. For inert bonds, like bronze, the diamond grain is held inside the bond due to positive locking. This leads to a linear correlation between grain concentration and critical bond stress [17]. The described phenomenon also allows the calculation of a maximal diamond concentration for the respective bond system. Further investigations have offered more correlations between the manufacturing process and the grinding application that can be quantified by critical bond stress [18]. Additionally, special single grain grinding tools were developed to allow the investigation of the interaction between the bond

matrix and a single diamond grain, which also provides a microscopic characterization of the interactions [19]. It was postulated that the additional, attractive interactions between bond and diamond grain will lead to higher critical bond stresses [17]. This work will expand upon this interaction between the metallic bond and diamonds by introducing carbide-forming elements.

2. Experimental

For the performed investigations, different kinds of grinding layer samples were manufactured. The reason for this is that entire grinding wheels are not suitable for some investigations or make the preparation of samples for mechanical testing quite complex. These samples have a cylindrical shape with a diameter of 22 mm and a height of approximately 5 mm. The height depends on the amount of bond and abrasive grains inside the sintering mold and the compression ratio. Therefore, deviations from the target height of 5 mm are a result of insufficient compacting, which eventually leads to porosity in the sample. The actual height of each sample was measured after sintering. Within one batch, there are seven specimens in the sintering mold, ensuring statistical reliability due to additional measurements in case of the application of destructive methods. All sintering processes were conducted with a Dr. Fritsch DSP 510 sintering press. The implemented control system allows the use of different sintering molds under similar conditions, and the monitoring system makes the identification of differences from the targeted parameters possible. Table 1 shows the used bond compositions the corresponding process parameters, sintering time (ts), sintering temperature (Ts), and sintering pressure (ps). Two series of investigations with different sintering parameters and bond compositions were performed. The composition Ti45 was used in both series. All experiments were performed in vacuo to prevent oxidization of the materials. The diamond grains used were blocky D251 grains from Ceratonia (CNF3060). A smaller grain size would have the advantage of a higher specific surface area, which can react with other components of the bond matrix and therefore gives a larger amount of reaction product. However, the utilized larger diamonds allow the characterization via optical microscopes. This makes it possible to observe changes to the condition of the grinding grain more easily. The metal powders for the bond where premixed Cu/Sn 80/20 (by mass) from Dr. Fritsch GmbH & Co.KG and different titanium and nickel powders from abcr GmbH that were directly mixed with the diamond grains. All mixing was done in individual HDPE containers inside a WAB Turbula 3D mixer. For each experiment, the powders were mixed individually, and under constant conditions with a total mixing time of 30 minutes for each batch and at 100 rpm. A cylindrical polymer container with a screw cap and a volume of 500 mL was used for mixing.

name	Ti	Ni	Ti	Ni	Ts	ps	ts
	at%	at%	wt%	wt%	К	МРа	S
Ti12	12	88	10	90	993 - 1,373	3,500	300 - 600
Ti30	30	70	26	70	993 - 1,373	3,500	300 - 600
Ti45	45	55	40	55	993 - 1,373	3,500	300 - 600
Ti45	45	55	40	55	993 - 1,093	2,500 - 3,500	480 - 960
Ti52	52	48	47	48	993 - 1,093	2,500 - 3,500	480 - 960
Ti62	62	38	57	38	993 - 1,093	2,500 - 3,500	480 - 960

Table 1. Compositions and Process Parameters of Performed Experiments

Mechanical characterization was performed via a three-point-flexural test of the cylindrical grinding layer specimen using a dynamometer The critical bond stress was calculated with the experiment's geometrical parameters length between supports l = 14 mm, sample diameter d = 22 mm and actual sample height h = 5

mm (measured for each sample). The obtained maximal force right before the point of fracture and the sample dimension, as well as the geometrical conditions of the measurement setup, were used to determine the critical bond stress following an already established method for cylindrical samples [16].

For the description of the reaction between the binder material and carbon (diamond), X-Ray diffraction (XRD) patterns were obtained by measuring the surface generated by the fracture in the three-point flexural test. This way an influence from the reaction between the binder and the graphite mold can be avoided. A diffractometer Seifert XRD 3003TT with a copper or a cobalt target was used to collect the data. The theoretical diffraction patterns used to evaluate the X-ray data, as well as the evaluation itself was done by the software PowderCell 2.3 [20].

Scanning electron microscope (SEM) micrographs were obtained via a Carl Zeiss EVO 60 XVP microscope. The backscattered electron detector (BSE) was used to characterize the surface generated by the fracture during mechanical testing, allowing the distinction between diamond grains (carbon) and the elements with a higher atomic number forming the bond (nickel, titanium, copper, tin).

3. Results and Discussion

3.1. Bond-Grain-Interface

Using a bond material that also contains a carbide-forming element, in theory, allows the formation of carbides in the interface of diamond grains and bond. The presence of titanium inside the binder material, for instance, introduces the possibility to form TiC. In general, the formation of carbides from elemental precursors usually requires high temperatures compared to the temperature range in which diamond is considered stable (<973 K). However, when sintering diamond tools the temperatures are commonly set below this upper limit to minimize thermal damages to the diamond. In order to investigate the formation of carbides, the temperatures are deliberately chosen to exceed the value of 700 °C in this study.

Due to the high melting point of pure titanium (1,941 K) compared to bronze (approx. 900 – 1,300 K), a binary system was chosen allowing the manufacturing of different bond matrices with different melting points and different chemical properties. For this paper, the binary system Ti/Ni was chosen whose phase diagram has a wide area with a lower melting point than pure titanium (<1,941 K). This pair forms a eutectic system with a minimal melting point at approximately 30 at% of nickel (Fig. 1). In principle, this also allows liquid phase sintering under reasonable temperatures that lessen the decomposition of diamond. Furthermore, this binary system forms a few intermetallic, crystalline phases compared to the bronze system (Cu/Sn), which additionally simplifies the phase analysis via XRD.



Fig. 1. Phase diagram of Ti/Ni [21], [22].

Because the reaction of diamond and titanium to titanium carbide ($Ti + C \rightarrow TiC$) can also be considered as "damage" to the diamond, a series of experiments was performed to determine the beginning of the formation of TiC during a timeframe that represents typical sintering processes of a grinding wheel (< 30 min). After sintering, the grinding layer specimens were mechanically tested, resulting in breakage along the area of highest stress concentration (Fig. 2; bottom right). These samples were then used as cross-sections for further preliminary investigations. Micrographs of three cross-sections are shown in Fig. 2. The differences in their appearance are quite noticeable. In the case of the sample containing the bronze bond (Cu/Sn: 80/20), distinct geometrical shapes can be seen on its surface. These shapes correspond with the diamonds of the grinding layer, however, only the shapes with a yellow tint are actual diamond grains (Fig. 2; 2). Shapes with a grey color (bronze with a higher tin content) or a slightly reddish tint (bronze with a lower tin content) are in fact impression inside the bond (Fig. 2; Fig. 3). The smooth parts are produced by the crystal faces from a diamond that is either in the corresponding location on the other part. The color in which the individual transferred faces appear is dictated by the bronze composition at the former interface. The Ni/Ti-bond system (Ti12) sintered at the same temperature exhibits the same principal surface features, yet the impressions are not as pronounced, and the diamond surface is slightly more opaque. Another batch with the same composition was sintered under significantly higher temperatures to force a reaction of the diamond with the surrounding bond. This leads to a substantial change in appearance, where the diamond grains are black and entirely opaque.



Fig. 2. Optical micrographs of grinding layer samples after the flexural test.

The XRD analysis of this sample (Ti12; 1,373 K) shows the presence of significant reflexions corresponding to TiC, as well as Ni, TiNi3, and the cubic phase of NiTi (Fig. 3). A diamond reflexion was not observed in this measurement, which is a result of the small probe area with an approximate diameter of 1-2 mm and the large, mostly monocrystalline diamonds. Due to the blocky character of the diamond grains, each grain can be regarded as mono-crystalline. Larger diamond grains mean a lower absolute number of grains in the grinding layer with a fixed grain concentration (V%). This leads to difficulties in observing diamond reflexions due to the low number of crystal orientations and therefore a low observation probability for the detector. However, due to their large size and characteristic color as well as transparency diamonds can be observed by an optical microscope. The utilization of XRD-investigations for this purpose is this not mandatory. In none of the measurements of the cross-sections, a reflexion of graphite was observed (graphite {100} $2 \, \Theta \, \approx \, 31^{\circ}$ for Co,k α). Even in cases where diamond reflexions were observed, no indication towards graphite could be found. The only reflexions of carbon-containing phases besides diamond were caused by titanium carbide.



Fig. 3. XRD analysis of grinding layer samples.

With the use of the backscattered electron detector (BSE) of an SEM (Fig. 4) the diamond grains (dark areas) can be easily differentiated from the bond (light areas). These high-resolution images present the grain surface in more detail and demonstrate that the diamond grains inside the bronze bond are in almost pristine condition. Grains in the Ti12-bond on, the other hand, have a rough surface with flattened edges. The rough surface coincides with a lighter shade of gray. This can be seen in areas with broken diamond grains, where there is a distinct difference between the dark surface of the cleaved surface (4) and the regular surface of the grains (3). Because the brightness in the image correlates with the atomic number, this lighter surface indicates heavier elements (Ti, Ni) on the surface. Together with the formation of TiC which could be observed via XRD analysis (Fig. 3), it is reasonable to conclude that there also is the formation of TiC in these cases. If there was no formation of TiC, the erosion of the diamond grains has to come from the formation of another carbon-containing compound. The formation of graphite would also explain this effect; however, the presence of graphite then should have also been observed via XRD, which was not the case. While there is no graphite present after the sintering process, it is possible that graphitization occurs during the process. The presence of graphite under these conditions can also produce TiC. This reaction, in turn, would consume the graphite in the process. However, the exact reaction route or mechanism has little influence on the sintering result. The process of carbide formation leads to the loss of the distinct crystal faces that are present in the diamonds before the sintering process.

An important difference between the Ti12-bond and the bronze bond is the fact that fractured diamonds are predominantly found in the Ti12 samples. These display a structure that can be described by the result of a conchoidal fracture, which is typical for fractures of some brittle materials like quartz (Fig. 4). Also, grains with a 'smooth' surface that are found in samples with the Ti12-bond, are exclusively fractured diamonds. Because for both samples at 993 K the sintering parameters like temperature, time and pressure are the same, this observation indicates that diamonds were fractured after the sintering process, which in turn can be a sign of improved grain retention in this bond system.



The fact that all tested grinding layer samples broke reasonably clean into two halves allows the side-byside comparison between both halves. Fig. 5 (top left) shows a micrograph of both halves of the NiTi; 993 K sample. The dotted rectangles indicate the part of the area that is used for the comparison, and the dotted cross is used as a reference. As one surface has to be rotated along the fracture line by 180° to reproduce the initial situation after the fracture, the image of one of the surfaces has to be mirrored along the vertical axis. This produces two images that can be overlapped. A comparison between the two halves produced by the mechanical test results in pairs of corresponding grains and indentations, and occasionally a pair of fractured diamonds (Fig. 5). In general, three different features are found on the fracture surfaces of the samples: a diamond grain on one of the halves and a corresponding impression on the other half (paired), fractured diamonds on both halves (fractured), or impressions on both halves (lost). The three examples illustrate that the ratio of these features varies depending on the bond composition and manufacturing conditions.



Fig. 5. Comparison of the surface on two corresponding fragments.

To simplify the evaluation of the respective surfaces, a scanning electron microscope was used. The grayscale images from the BSE detector allow an easier differentiation between bond and diamond. Same as with the images of Fig. 4 areas with carbon appear darker and areas with titanium or nickel appear brighter. An approach similar to the images from the optical microscope leads to an improved overview of the surface caused by the fracture: Fig. 6 shows the images of the right half (R), the left half, but mirrored across a vertical

plane (L-m), and both images overlapped and multiplied (overlay-mult.). The multiplication can be understood as the numerical multiplication of the brightness value of each pixel of the overlay. This leads to more pronounced light and dark values, where these extreme values occur on both images. This means that the darkest values on the overlay are generated by areas where diamonds are on both halves. Generally, this can be caused by two circumstances: either there is a cluster of diamonds split by the fracture area, or a diamond was fractured during the mechanical test and each part remained inside the bond. Because the diamonds outer surface is brighter when they reacted with the bond, fractured diamonds appear even darker in the case of the Ni/Ti-based bond. In a color-mapping process the image is transformed so that the very dark areas appear red, the dark areas yellow, and everything else grey. This facilitates the distinction between grains that are paired with an impression, and grains that are fractured or part of a cluster. The distinction between the latter two is not possible by the color alone, however, color mapped images aid the characterization of the sintering result (Fig. 6: whole fractured surface).

This achieves simplified images for the characterization of the grain on a qualitative level. The removal of the textures of the bond and the classification of grains into red and yellow help in the characterization of large areas, ultimately making it possible to generate characteristic values for the comparison between different grinding layers. Different values, like area ratios of the different colors, and the number of counts are being investigated for this purpose.



Fig. 6. Image processing of SEM micrographs to generate a color map.

The comparison between larger areas of the three different samples illustrates how the samples differ in several aspects. Grains of the Ti12 samples, for example, have less defined shapes and edges than grains of the bronze sample (Fig. 7). Another difference between both systems is that the number of red areas is considerably higher for the Ti12 samples. When considering the whole fractured area the color map a ratio of red pixels to all pixels can be calculated. This ratio differs significantly between the three samples with 0.9% for the bronze sample, 2.9% for the Ti12 sample sintered at 993 K, and 2.1% for the Ti12 sample sintered at 1,373 K. Even though the brightness values that form the basis for the color map are defined in a subjective manner, the different ratios capture the differences exhibited by the respective surfaces very well. Upon closer

inspection, it becomes apparent that red areas on the surface of the bronze sample are mostly caused by overlapping grains in clusters. These have to be checked manually; however, the number of red areas for bronze samples is significantly lower than for the Ti12 samples and fractured grains are virtually exclusive to the Ti12-system. While it may be possible that the sintering process at 1,373 K caused additional thermal damage to the diamond grains, what in turn would lead to a higher number of fractured grains, it cannot explain the difference between the 993 K sample of Ti12 and bronze, as both are sintered under the same conditions.



Fig. 7. Color maps of cross-sections of three grinding layer samples. L: left side; R: right side; O: overlay.

Another difference between both systems can be illustrated by the yellow values of the color map. The apparent loss of defined edges of the diamond grains when comparing the Ti12 samples to the bronze sample also shows in the ratio of yellow pixels to all pixels. The respective ratios differ significantly between 47% for bronze, 29% for Ti12; 993 K, and 34% for Ti12; 1,373 K. This indicates, that there is a difference between grain at the surface and visible grain at the surface, which is expected due to the reactions, and the bond adhesions at the surface of the diamond grains in the Ni/Ti bond.



Fig. 8. Strongest effects on the critical bond stress.

An indication for the progress of the sintering process is the formation of new phases corresponding to the respective points in the phase diagram (Fig. 1). Due to the formation of crystalline phases, the transition can be observed via XRD. Because of this, diffractograms were measured to aid in the analysis of the correlation of sintering parameters and critical bond stress. In SEM micrographs, the granular structure of the bond matrix resembles more a loose bulk, than a homogeneous metal bond. For the three compositions at Ts = 993 K, and ts = 480 s the diffractogram primarily consists of isolated nickel and titanium phases (Fig. 9). After increasing the parameters to Ts = 1,093 K and ts = 960 s a significant formation of the expected intermetallic compounds Ti2Ni and Ni3Ti was detected. In general, these samples also exhibited higher critical bond stresses, with an increase by a factor of two. Because the statistical DOE approach changed several parameters at a time, a separate set of experiments were conducted, specifically to examine the influence of the temperature and the presence of diamond grains.



Fig. 9. X-ray diffraction pattern (Cu k α) of different grinding layer samples.

The introduction of diamond grains into a bond system leads to significant differences in the mechanical behavior of the grinding layer, depending on the grain concentration. This change directly impacts critical bond stress. The effect can be explained by the fact that an increase in grain concentration always leads to a volumetric decrease in the bond material. If there is no attractive interaction between diamond and bond the grain is solely held via a form fit. For the grinding layer, an increase in grain concentration results in a decrease in critical bond stress. Previous work shows this effect in the bronze system, describing a linear downward trend of the critical bond stress with increasing grain concentration. Furthermore, this linear correlation gives a theoretical maximum grain concentration. [17] Fig. 10 shows critical bond stress values of several different grinding layer samples, each bar being aggregated from the measurement of seven individual samples. In the case of the 80/20 bronze, the decrease in critical bond stress due to the presence of 25 V% diamond is visible (-40%). The analysis of corresponding samples from grinding layers with nickel and diamond, and titanium and diamond result in higher values, indicating a more resilient grinding layer in both cases. The example of Ti45 (right graph) illustrates that the pure bond has higher critical bond stress, as it was expected. An increase in temperature does not affect the value significantly, indicating that the bond has reached a point at which the initial bond grains are sintered together in a way that results in a good cohesion between these particles. However, for samples with 25 V% diamond, this temperature difference results in a change of the critical bond stress. An increase in temperature of 100 K leads to an increase in critical bond stress of approximately 40%, which indicates that there are additional effects present. This is most certainly due to chemical reactions between the bond and diamond grains. Further increasing of the temperature seems to negate this effect in part. This may be explained by excessive reactions of the diamond, which ultimately damages the diamond grains and decreases the interaction between grain and bond and in turn reduces the overall cohesion inside the grinding layer.



Fig. 10. Critical bond stress of samples with different bond compositions, with, and without diamond grain.

A detailed look at the Ni/Ti-bond system demonstrates a more complex relation between critical bond stress and temperature depending on the bond composition (Fig. 11). Ti12, for example, shows an additional increase in critical bond stress for the samples without diamond grain. This highlights the fact that due to the high nickel content, this bond composition needs higher sintering temperatures to generate reasonable cohesion inside the sintering sample, indicating that the sintering result has not yet reached a stationary point. This, in turn, means that an increase in critical bond stress for samples with diamond most likely includes, but is not necessarily restricted to, the formation of a tougher bond matrix. While the critical bond stress of the samples without grain increase by roughly 30% the increase for the samples with diamond is 40%. If there was no additional interaction between bond and grain, it should be possible to describe the relationship between grain concentration and critical bond stress with the linear behavior found in the bronze system from previous investigations. [17] In this case, plotting the critical bond stress versus the grain concentration would show a linear downward trend ultimately crossing the x-axis at a certain point, indicating the theoretical maximum grain concentration. Assuming this trend to be true for this case, the two values (C = 0 V% and C = 25 V%) a linear fit crosses the abscissa at approximately 70 V%. The comparatively high increase in critical bond stress for the samples of Ti12 with diamond grain with increasing temperature is an indication for the presence of additional interactions between bond and diamond grains. This observation also corresponds well with the color maps in Fig. 7, supporting the general conclusion of additional attractive interactions between bond and diamond grain. However, for a definitive answer, more samples at higher sintering temperatures have to be investigated. For these investigations, an additional focus has to be set on the analysis of the thermal damage to the diamond grain, as it will most likely take place to a higher degree and cause an opposing effect.

For Ti30 the phenomenon of improved critical bond stress is not present, as it was with Ti12 and Ti45. While there is a slight increase in critical bond stress with increasing sintering temperature in the samples without diamond grains, the critical bond stress of samples with diamond seem to stagnate. This shows that the interactions between bond and diamond depend on more than just the titanium content. The fact that the

most consistent increase of critical bond stress occurs in the samples with the lowest titanium content implies the presence of an interplay between other factors. This could be caused by a different chemical environment due to the presence of larger amounts of TiNi3. A definite answer to the reason for this behavior, however, requires a closer investigation of the chemical reactions of this system. Another explanation can be for example the wettability of the diamonds' surfaces, or increased specific surface areas due to erosion of the diamond. It is also possible that reactions at the interface between diamond and bond have progressed so far, that the volume of the interface has grown to a point that the problem of the missing chemical retention is pushed further away from the grain. These aspects put together warrant further investigations into the specifics of the variation of the critical bond stress.



Fig. 11. Critical bond stress of samples with lower titanium contents, with, and without diamond grain.

4. Conclusion

When compared to inert bond systems, like bronze, the active bond system of titanium and nickel deviates from the expected behavior, when increasing the grain concentration. It could be shown that it is possible to use a bond from nickel and titanium for diamond grinding layers. However, all samples showed higher critical bond stresses than the bronze grinding layer, which was used as a reference, indicating a stronger cohesion inside the grinding layer. In the case of a combination of 25 V% diamond in a bond consisting of Ti45, increasing the temperature also increases the critical bond stress compared to a sample manufactured at the same conditions without diamond grains. At 993 K the sample containing diamonds exhibits 59% of the critical bond stress of the reference sample at 993 K. Increasing the temperature by 100 K improves the relative critical bond stress to 82% of the sample without diamond grains. Similarly, Ti12 also displays an increase in relative critical bond stress from 51% to 62%. However, in this case, the overall absolute critical bond stress also increases. Yet this is not true for all compositions. While Ti30 shows higher critical bond stress than the bronze system, the beneficial effect of the active bond at higher temperatures could not be observed, further illustrating the complexity of the interactions of a rather 'simple parameter' like bond composition. The investigation also shows that the analysis of critical bond stress allows the evaluation of the completion of the sintering process. When the temperature is increased and at the same time, the critical bond stress of pure bond stays constant, it can be assumed that a stationary point is reached. This allows the differentiation of increases in critical bond stress due to interactions between bond and grain and changes due to differences in the bond matrix itself.

The reflexions of titanium carbide can be seen in XRD analyses to varying degrees, and prove the formation of titanium carbide at the interface of diamond and bond. SEM and microscope micrographs underline this

observation. The degree of this reaction depends on both, the composition of the bond and sintering temperature/time. Optical and SEM analyses of the cross-section after mechanical testing can be used to characterize the bond concerning the diamond grain and its retention. Grain outbreaks and fractured diamonds can be differentiated from grain/impression pairs, allowing the evaluation of the grain retention in a semi-quantitative manner. In combination, these methods can be utilized to characterize the bond matrix, the diamond grain, and the grinding layer as a whole for systems with active bond materials. This approach can be used for the evaluation of a whole set of manufacturing parameters without having to build an entire grinding wheel.

This approach may be especially helpful for the development of future bond systems for grinding layers with a reduced form fit, like for example in 3D-printed structures. Besides the nickel-titanium system, 'active bonds' with other carbide forming elements, like chromium, for example, should be investigated to derive more general connections between carbide grain-bond interfaces and the resulting grinding behavior of the corresponding tools. Furthermore, these systems should also be investigated in a systematic approach on a microscopic level to gain a more fundamental understanding of the exact interactions. In such an investigation, a comparison of carbide interfaces that are formed during the manufacturing process and those formed beforehand, as it is the case with TiC-coated diamonds, for example, should take place. This research would close a gap in knowledge between the material side in which these reactions are already well understood and the application of these materials that are often affected by different restrictions, such as the applicability of reaction conditions or economic constraints.

Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

Berend Denkena: Funding acquisition, Project administration, Supervision, Writing - review & editing, Resources. Alexander Krödel-Worbes: Project administration, Writing - review & editing, Validation, Conceptualization. Fabian Leander Kempf: Visualization, Writing - review & editing, Writing - original draft, Data curation, Investigation, Formal analysis, Methodology. All authors have approved the final version.

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Berend Denkena is the head of the Institute of Production Engineering and Machine Tools at the Leibniz Universität Hannover.

After obtaining the doctorate from the Faculty of Mechanical Engineering at the Leibniz Universität Hannover in 1992, he worked as a design engineer and head of various research groups for Thyssen Production Systems both in Germany and the United States. From 1996 to 2001 he was the head of Engineering and Turning Machine Development at Gildemeister Drehmaschinen in Bielefeld. Since 2001 he has been a full professor of

Production Engineering and Machine Tools and director of the Institute of Production Engineering and Machine Tools at the Leibniz Universität Hannover.

Prof. Denkena is a CIRP fellow member. His primary areas of research are geometry and functionalizing manufacturing processes, machine tools for cutting and grinding, production planning and control, and simulation of manufacturing processes.



Alexander Krödel-Worbes is the head of Department "manufacturing processes" of the Institute of Production Engineering and Machine Tools at the Leibniz Universität Hannover, where he is responsible for over 30 researcher engineers. In 2014, he joined the IFW in the Department of Manufacturing Processes. He was responsible for the group "cutting technologies" from 2016.



Fabian Leander Kempf is a research assistant of the Institute of Production Engineering and Machine Tools at the Leibniz Universität Hannover since 2016. From 2012 to 2015 he was a research assistant of the Institute of Inorganic Chemistry at the Leibniz Universität Hannover in the work group of Prof. Dr. Peter Behrens, researching metal-organic frameworks (MOF), and related materials as part of his doctoral thesis. His research activities are focused on the manufacturing of grinding tools.