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Fabrication and use of Cu-Cr-diamond composites for the application in deep feed grinding of tungsten carbide

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B. Denkena, A. Krödel, R. Lang^{*}

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

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ABSTRACT

Keywords: Carbides Composites Synthetic diamond High pressure high temperature (HTHP) Abrasion Wear Interface characterization Mechanical properties characterization Thermal properties Cutting tools

Machining of tungsten carbide requires the use of highly wear resistant grinding tools, like metal bonded grinding tools. The abrasive layer of these grinding tools can be regarded as Metal-Matrix-Composites reinforced with diamond particles. Copper-Matrix-Composites already are being used as heat sink materials through their outstanding high thermal conductivity. In this work, Cu/Diamond composites with 50 vol% diamond have been fabricated through field assisted sintering and the application of these composites as grinding layers in a deep feed grinding process of tungsten carbide was investigated. Through addition of chromium powder as a carbide former on the surface of the diamond particles, the critical bond strength and therefore the diamond grain retention was significantly increased by +363%. The addition of 2 wt% chromium to the copper matrix also resulted in a +84% increase of thermal conductivity relatively to the chromium free Cu/Diamond composite. Grinding of tungsten carbide as a dynamic stress test showed that the increased grain retention and thermal conductivity and the formation of adhesive cloggings on the grinding wheel surface during grinding.

1. Introduction

Metal bonded diamond grinding wheels are frequently used in high efficiency and precision grinding of hard to cut materials like tungsten carbide or cermet. Characteristics of bronze bonded superabrasive grinding wheels are a high bonding strength, high thermal resistance and a long tool life [1]. Bonding systems for metal bonded diamond grinding wheels consist in most cases of copper-tin bronze. Besides abrasive grain size, type and concentration, the bond material has major influence on the key properties of the grinding wheeling including wear mechanisms and thermal conditions in the wheel-workpiece contact zone [1]. The bonding material also affects the grain retention of diamond in the metal matrix. One of the main technological difficulties in designing grinding wheels is controlling the amount of grain retention, so that the diamond grains are not released to early from the bonding matrix, but also do not become flattened due to long use during grinding [2]. Tungsten carbide often finds application for cutting and drilling tools because of its high wear resistance, high flexural strength and hardness. Through the grinding process the hard and brittle material can be influenced by deformation, microcracking or residual stresses that can reduce the surface integrity of the material [3]. However, introduced compressive residual stresses can even improve flexural strength of the materials [4]. Reducing thermal load while applying same mechanical load during grinding can result in an increase of introduction of compression instead of tensile strength and therefore improve the flexural strength and wear resistance of cutting and grinding tools made from tungsten carbide [5].

Recent research originally inspired by developments in microelectronics and the need of material with high thermal conductivity (TC) advanced the knowledge about metal matrix composites reinforced with diamond particles sintered with high temperatures techniques. Especially, Copper-Matrix-Composites reinforced with diamond particles have gained interest, due to their outstanding thermal and good mechanical properties. Besides the fabrication method of field assisted sintering technology (FAST) that is used in the present study there is a broad field of fabrication methods like: gas pressure infiltration [6–8], electroplating [9,10] or metal infiltration [11,12] However, because of the naturally non-wetting behavior between diamond and Cu it is difficult to obtain good interfacial bonding and a high Interfacial Thermal Conductance (ITC) that directly influences the TC and the grain

* Corresponding author. *E-mail addresses:* denkena@ifw.uni-hannover.de (B. Denkena), kroedel@ifw.uni-hannover.de (A. Krödel), lang@ifw.uni-hannover.de (R. Lang).

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retention [13]. An approach to overcome the inert behavior between diamond and Cu is doping with elements that can lead to carbide formation within the interface such as Cr [14], Ti [15], B [16] or W [17]. These elements can be used as coating material on the diamond particles prior to the sintering process [7] or as powder additive to react in-situ [18]. The latter not only has an effect on the diamond/metal interface, but also effects the thermo-mechanical properties of metal matrix. The formation of carbides can not only increase the ITC but also the grain retention of diamond in the Cu matrix through formation of the interfacial phase between Cu and diamond. Using chromium powder as an additive while sintering (Cu-Cr)-50 vol% diamond composites a TC of 518–658 W m⁻¹ K⁻¹ was obtained by multiple researchers [18,19]. [20] reported that the thermo-physical properties are strongly influenced by the amount of added Cr and the resulting thickness of the interfacial carbide layer. The maximum of TC is reached at lower chromium contents then is needed for reaching the maximum of mechanical strength and therefore an increase of grain retention [21]. Emerging interfacial phases while sintering with chromium additive can be identified as the brittle chromium carbide phase Cr₃C₂ [22], Cr₃C₇ or $Cr_{23}C_6$ [23]. When sintering with additives that have a reactivity with carbon there is a risk of graphitization of the diamond. Chromium shows a high reactivity towards carbon, but effects of graphitization within the interface were not reported, making chromium a promising additive for the increase in interfacial adhesion through carbide formation [24]. The application of Cu-Diamond composites that are not related to brass or bronze as grinding tools is described little in the literature. [25] designed a fabrication process and studied the effect on fabrication conditions of a pure copper grinding wheel by centrifugal sintering casting method for machining of carbon fiber-reinforced plastic. [26] used Cu-Ti and Cu-TiH2 as additives for the fabrication of the Cu-Diamond composites in an hot pressing process at 720 $^\circ\text{C}.$ Tribological tests were carried out on the fabricated composites resulting in a tenfold reduced wear of the Cu-TiH2 -Diamond composites relatively to a pure Cu-Diamond composite.

In this work, copper-matrix-composites were sintered with chromium as an additive for the application as grinding wheels in deep feed grinding of tungsten carbide. The composites were characterized regarding their thermal and mechanical properties, as well as the emerging phases due to the reaction of chromium and diamond was investigated.

2. Experimental procedure

To investigate the influence of chromium addition on the material properties, bond composition and carbide formation cylindrical abrasive layer specimens with a diameter of 22 mm have been fabricated by the Field Assisted Sintering Technology (FAST) using a Dr. Fritsch GmbH & Co. KG DSP 510 sintering press in vacuum. Dr. Fritsch GmbH & Co. KG DIACU4500 copper powder with an particle size of <45 µm, Ceratonia GmbH & Co. KG CNF3080 diamond powder (46 µm) and ABCR GmbH chromium powder with an average particle size of 10 µm was used. Powder mixtures of 50 vol% diamond and 50 vol% bond mixture of copper and varying amounts of chromium were given to a Turbula T2F powder blender mixer for 30 min at a cycle rate of 101 1/min. Afterwards, the mixture was consolidated in a graphite die, pressed at a load of 35 MPa and sintered with a heating rate of 100 °C/min and a holding step at 900 °C for 1200 s. The chromium content of the bond mixture has been varied in the following steps: 0; 0,1; 0,2; 0,5; 1; 2; 4; 8 wt%. Hereinafter, the bond compositions will be labeled as Cu-xCr with the index x as the chromium content in wt%.

All specimens were uniaxial loaded until they collapsed in a 3-point flexural test to explore the mechanical properties of the composites. The vertical force Fz was measured with a Kistler dynamometer 9255B and afterwards the critical bond strength can be calculated using the distance between the two mechanical bearings underneath the specimen l, the diameter d and the height h of the specimen. Assuming that the area of moment inertia is $I_y = (b \cdot h^3) / 12$ from beam theory the critical bond strength CBS can be calculated after:

$$\sigma = \frac{3^*Fz^*l}{2^*d^*h^2}$$

The density of the specimens was measured based on Archimedes' principle using an EMB 200–3 V density scale from Kern & Sohn GmbH.

The fractured surface of the collapsed Cu-xCr specimens and the morphology of the carbide phase were observed by scanning electron microcopy (SEM) with a Joel JSM-7610FPlus. X-Ray diffraction patterns in a range of 20° -99,95° with step length of $0,05^{\circ}$, were obtained by a two-circle diffractometer system XRD 3003 TT in a Θ/Θ setup, using Cu Ka radiation.

The thermal conductivity (TC) λ of the composites at 23 °C has been calculated following formula (1).

$$\lambda = a^* p^* c_p \tag{1}$$

where a is the thermal diffusivity, p the density measured by the Archimedes' principle and c_p the specific heat capacity. The thermal diffusivity was measured by a laser flash apparatus (LFA 447, Netzsch) using disk-shaped samples of 12.7 mm \times 3 mm. The specific heat capacity of copper, chromium and diamond was measured separately by differential scanning calorimetry and the specific heat of the composite material was calculated through the rule of mixture based on the mass fraction of each component.

To explore the behavior of the composite material under dynamic stress in the application of grinding, 1A1 grinding wheels with a diameter of 100 mm and a width of 10 mm were sintered under the same conditions as the specimens were sintered before (900 °C for 1200 s). Diamond concentration has been set to 50 vol% while three different chromium contents have been used: 0, 2 and 8 wt%. To achieve equal initial grinding tool topographies the grinding layers are dressed by means of a #70-SiC form roll (q_d = -0.8; v_{cd} = 9.3 m/s; v_{fad} = 800 mm/ min; $a_{ed} = 0.2$ mm) and sharpened with a Saint Gobain Nr.5 sharpening stone ($a_{es} = 3 \text{ mm}$; $v_{fs} = 100 \text{ mm/min}$; $v_{cs} = 18 \text{ m/s}$). The dynamic stress test is carried out by a deep feed grinding of tungsten carbide on a Geibel+Hotz FS 840 KT surface grinding machine. To observe differences of the grinding behavior of the grinding tools a sufficiently high specific material removal of $V'_w = 2500 \text{ mm}^3/\text{mm}$ is machined by each grinding tool. The experiments are carried out with a width of cut $a_p = 4$ mm, so that a part of the grinding layer does not engage during grinding. In this way the initial tool topography and diameter can be compared with the topography and diameter of the wear zone that is engaged during grinding. Grinding wheel surface topography is obtained by means of confocal microscopy using a Confovis DUO Vario and is evaluated for radial tool wear Δr by comparison of the average diameter of the area of grinding wheel that is engaged in the grinding process (wear zone) and the average diameter of the grinding wheel area that is not engaged during grinding (Fig. 1).

3. Results and discussion

3.1. Influence of chromium addition

The critical bond strength (CBS) was used to characterize the macroscopic mechanical behavior of the composites. Fig. 2 shows the CBS data of the Cu-*x*Cr specimens containing 50 vol% diamond. The addition of chromium results in a drastic increase of CBS up to 505 N/mm² at a chromium content of 2 wt%, which represents an increase of +363% compared to the CBS at 0 wt% chromium of 136 N/mm². The increase of chromium content above 2 wt% results in a decrease of CBS to 423 N/mm² at 8 wt% chromium (Fig. 2). This is related to the increase of Cr precipitates and undissolved Cr particles in the copper matrix with increasing chromium addition and the resulting increase in hardness of the Cu-*x*Cr matrix (Fig. 3). The growing content of Cr precipitates



Fig. 1. Schematic experimental setup and the grinding wheel after grinding including the wear zone.



Fig. 2. Change in critical bond strength of the composites fabricated by FAST sintering with increasing chromium content.

increases the precipitation hardening effect due to the hindrance of dislocation movement resulting in a decrease of ductility. The measured density of the composites peaks at 0.1 wt% chromium and is increased in comparison with the Cr-free specimens (Fig. 4). The increase in density is caused by the closure of gaps and voids in the diamond-metal interface trough initial carbide formation. With further chromium addition, the density decreases due to the lower density of chromium compared to the density of copper.

SEM micrographs of the fracture surface of the composite specimens show insight into the in-situ carbide formation at the surface of the diamonds and the morphology of the bonding matrix. Without addition of chromium no carbide formation is observable. Imprints of diamond outbreaks indicates segregation of diamonds from the Cr-free Cu-matrix and the low interfacial adhesion (Fig. 5a). At Cu-0.1Cr minor carbide formation covering only few percentages of the diamond surface is observable. Further addition of chromium to 0.2; 0.5 and 1 wt% results in an increase of partly covered surface area by the in-situ formed carbide. A complete coverage can first be observed at 2 wt% chromium, while some diamonds tend to break internally due to the load that is applied during 3-point flexural test. This indicates the strong interfacial adhesion that is in accordance with the CBS data (Fig. 5c) Fig. 5d shows that the broken diamond grains are characterized by an interfacial carbide layer between diamond and the surrounding CuCr-matrix with an unaffected fractured surface. The thickness of the interfacial carbide



Fig. 3. a) Images of the polished Cu-xCr specimen without diamond grains and varying contents of chromium. b) Change of vickers hardness for the Cu-xCr alloy versus chromium content.



Fig. 4. Change in density of the composites fabricated by FAST sintering with increasing chromium content.

layer is estimated with less than 1 μ m but further research is necessary to give an exact value. Additional increase in chromium content to 4 and 8 wt% does not significantly change the morphology of the fractured surface, resulting in the assumption that local availability of Cr has a major influence on the coverage of diamond with chromium carbide. The diffusion coefficient of Cr in Cu at 900 °C is generally low with a value of $65.5 \cdot 10^{-15}$ m/s [27]. Cr particles that cannot diffuse far enough to react with diamond remain undissolved in the Cu matrix. Furthermore, the solubility of Cr in Cu at 900 °C is, in accordance with various researchers, below 0.5 wt% and decreases with decreasing temperature, so that excess chromium can also remain as precipitates in



Fig. 5. EDX-Mapping of the fractured composite surface with a), b) 0 wt% chromium addition and c), d) 2 wt% chromium addition. a) Grain imprints of diamonds and unaffected diamonds (grey) are visible within the copper matrix (blue) of the fracture surface. c) Chromium (green) accumulates as a layer surrounding the diamond grains from which some diamond grains are broken within the fracture surface. d) Detailed view of the chromium layer within the interface of a broken diamond grain and the copper matrix. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

the Cu matrix [28]. This is supported by the findings of increasing number of Cr precipitates or Cr particles and the slight decrease of CBS from 2 wt% to 8 wt% chromium addition through the embrittlement of the copper matrix.

3.2. Phase analysis of interfacial carbides

Diamonds have been extracted from the Cu-2Cr-matrix using nitric acid before XRD-analysis (Fig. 6). This chemical leeching procedure leads to the solution of copper but does not influence carbon containing crystallites [24]. In this way, the in-situ carbide coating and diamonds can be measured with relatively high intensity due to the absence of

copper. Through comparison and the good agreement of the theoretical reflections of Cr_3C_2 and the pattern of the released diamonds the formation of Cr_3C_2 can be emphasized. Furthermore, remnant Cu can be detected indicating an incomplete dissolution through the extraction process. The results verify the formation of the intended chromium carbide layer on the surface of the diamond grains.

3.3. Thermal conductivity

Prior to calculation of the TC shown in Fig. 7 the specific heat capacity c_p has been determined for the used chromium powder, the copper powder and the diamond powder by differential scanning



Fig. 6. X-ray diffraction patterns of diamonds that have been chemically released from a Cu matrix with 2 wt% chromium addition.



Fig. 7. The by the rule of mixture calculated thermal conductivity of the composites fabricated by field assisted sintering with various amounts of added chromium.

calorimetry. By the rule of mixture calculated data for the specific heat capacity of the composite materials are shown in Table 1. TC is increase by +84% when adding 2 wt% chromium to the copper matrix compared to the chromium free composite. Through the formation of the carbide layer an increase of ITC can be assumed. However, values above the theoretical TC of pure copper (400 W/m·K) have not been reached. A possible explanation for this is that the carbide layer thickness is strongly affecting the ITC. A discontinuous but thin layer can increase the ITC further than a thick continuous layer. The thin layer can enable better coupling of electrons that dominate the heat transfer in metals with photons that dominate the heat transfer in diamond. Increasing carbide layer thickness can result in a renewed increase of interfacial thermal resistance through the lower initial thermal conductivity of chromium [21]. A further increase of chromium content to 8 wt% leads to a decrease of TC. It can be assumed that the decrease is caused by an increase of undissolved chromium within in the copper matrix, which is also the case for the decrease of CBS mentioned above.

3.4. Grinding behavior

The wear behavior of grinding tools with various chromium contents has been characterized. Fig. 8 shows the grinding ratio (G-ratio) for the tools that have undergone the dynamic stress test. The G-ratio is defined as the volume of material removed by grinding divided by the volume of wheel wear. Without the addition of chromium, grinding resulted in a massive outbreak of grinding layer material, leaving the grinding wheel nonfunctional. G-ratio is marked here as tool failure. For the grinding wheel with addition of 2 wt% chromium a radial wear (Δr) of 18.6 μm after grinding of 2500 mm³/mm was detected resulting in a G-Ratio of 428. A further increase to 8 wt% chromium results in a decrease of G-Ratio to 154 with a radial wear of 51.8 µm. Fig. 8 also shows tool topography after grinding of 2500mm³/mm, showing that adhesive cloggings of the workpiece material tungsten carbide remain on the grinding wheel surface with a chromium content of 8% after the grinding process. On the surface of the tool with 2 wt% chromium addition these adhesive cloggings were not identified. A possible explanation for the lack of adhesive cloggings can be the higher thermal conductivity of the grinding layer material. It can be emphasized that this leads to a better heat removal from the contact zone between grinding tool and workpiece towards the grinding layer. This would result in lower workpiece temperatures and therefore prevents the formation of adhesive cloggings on the grinding tool surface.

4. Conclusions

Copper-matrix-composites reinforced with 50 vol% diamond particles have successfully been manufactured by field assisted sintering technique. An increase in diamond grain retention and thermal conductivity has been achieved through powder addition of 2 wt% chromium to the copper matrix. Thereby, an in-situ Cr3C2 layer formed between the whole diamond grain and the surrounding CuCr-matrix. Through precipitation hardening of chromium that does not take part in the formation of Cr₃C₂ the matrix material becomes more brittle with increasing chromium content. Furthermore, the excess chromium within in the copper matrix leads to a decrease of thermal conductivity compared to the composite material at 2 wt% chromium. Using the Cu (Cr)/Diamond composites as grinding layers during deep feed grinding of tungsten carbide it is evident that the addition of chromium has a significant influence on the tool wear. Using pure copper as bonding matrix resulted in a tool failure. Here, the low critical bonding strength and the relatively high ductility are not sufficient to withstand the forces during grinding and no continuous wear occurs. Addition of 2 wt% chromium leads to a continuous wear during grinding with a G-Ratio of 428.

Table 1

Input data for the calculation if the thermal conductivity of the Cu-xCr composites with 50 vol% diamond content.

Sample	Specific heat capacity cp $(J \cdot kg^{-1} \cdot K^{-1})$	Density <i>p</i> (g/ cm ³)	Thermal diffusivity a (m ² /s)
Cu-0Cr Cu-2Cr	0.320 ± 0.013 0.322 ± 0.014	$\begin{array}{c} 5.89 \pm 0.02 \\ 5.84 \pm 0.01 \\ 5.72 \pm 0.02 \end{array}$	97.8 ± 4.4 183.7 ± 5.1 150.1 ± 2.2
Cu-8Cr	0.330 ± 0.012	5.73 ± 0.02	150.1 ± 3.3



Fig. 8. G-Ratios of the grinding wheel with various chromium contents and the corresponding grinding wheel surfaces after grinding of a volume 2500 $\rm mm^3/mm$.

Credit author statement

Berend Denkena: Supervision. Alexander Krödel: Writing- Reviewing and Editing. Roman Lang: Conceptualization, Methodology, Original draft preparation, Visualization.

Prime novelty statement

This research paper describes for the first time the fabrication of Cu-Cr-Diamond composites with FAST for the application as grinding tools during deep feed grinding of tungsten carbide.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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