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Alumina composites with solid lubricant content

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Materials

ABSTRACT

Purpose: The aim of the study presented in this article is to determine the effect of the addition of solid lubricating substances on the selected properties of ceramic tool materials. The effect of tested lubricant additives on the density, porosity, Young's modulus and Vickers hardness of Al_2O_3-based ceramics with Ti(C,N) was determined.
Design/methodology/approach: Ceramic materials based on aluminium oxide with the addition of solid lubricating substances. Materials were obtained by uniaxial compression with the addition of sliding substances, then isostatically pressed, and dried. The sintering process was carried out at a temperature of 1750°C under high vacuum conditions. Density, Vickers hardness and Young's modulus was determined for these materials. The materials were also subjected to tribological analysis. The results obtained were compared with the properties of ceramic material based on Al_2O_3 + 30 mass.% Ti(C,N), from which tool materials were made.
Findings: The use of solid lubricating substances in the production of ceramic materials allowed a reduction in the coefficient of friction. Depending on the sliding additives used, the relative density values of individual materials were in the range of 85.3% to 92.3%. Young's modulus values for these materials were in the range of 273 GPa to 343 GPa, and Vickers hardness in the range of 966 to 1373 HV1. The results of tribological analysis showed a coefficient of friction value for the base material of 0.51. For the other materials, the coefficient of friction values were in the range 0.28 to 0.41, the lowest value being for the material with the addition of $BaZrO_3$.
Practical implications: Ceramic materials with the addition of solid lubricating substances may be used in cutting tools. Study of the composition and production technology of such tools allows for the minimisation of the use of liquid cooling lubricants in the machining process and the achievement of higher cutting speeds.
Originality/value: Ceramic materials intended for cutting tool blades were produced with the addition of materials of a layered structure or of high cleavage by free sintering. These materials were added to the structure, resulting in a reduced coefficient of friction, which was measured at between 55% and 75% of the coefficient of friction of the base material. The coefficient of friction was determined for friction pairs: ceramic material - high hardness steel ball.
Keywords: Ceramics; Solid lubricant; Alumina; Titanium carbonitride; Mechanical properties

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1. Introduction

Carrying out the machining process without the use of liquid cooling lubricants results in a reduction in investment costs and production costs. A reduction in friction and the elimination of fluids from the production process can be achieved by coating of the tool surface or by adding to the structure of the tool solid lubricating substances resistant to the action of high temperatures. The application of a coating on tools to reduce abrasive wear allows cutting to be carried out at high cutting speeds. There have been numerous studies concerning the synthesis, on the surface of cutting tools, of hard coatings based on nitride or titanium carbide [1-4].

Another approach is the synthesis, on the surface of cutting tools, of sliding layers or the addition to the structure of the tool materials of solid substances. These substances, during cutting, should create on the surface of the tool material a thin lubricating film. The principle of lubrication using solid sliding agents is based on the existence of easy-slip planes in the crystal structure. In the case of sliding materials, it is not sufficient that the material have a layered crystal structure. Prerequisite is the existence in such a structure of weak bonds between the layers [5, 6].

One of the longest established and best known lubricating materials is graphite. The coefficient of friction value of graphite on a clean surface in air ranges from 0.10 to 0.15. It should be noted that the coefficient of friction of graphite is strongly dependent on its environment. Above 500°C, oxidation takes place and the coefficient of friction value increases significantly.

Another well-known and commonly used lubricating agent is molybdenum disulphide which, like graphite, has a layered structure and is characterised by a low coefficient of friction (0.1-0.2). The low coefficient of friction of MoS_2 is maintained up to 200°C, after which it increases as a consequence of the emergence of an MoO_3 phase. This process occurs strongly above 400°C [7]. In the case of a WS_2-based sliding phase, decomposition occurs at a little higher a temperature (approx. 450°C), whilst complete oxidation does not occur even when the temperature is raised to 600°C. The decomposition product of the WS_2 phase is a WO_3 oxide phase, a sulphur (S) and SO_4^{2-} ions. In terms of stability under oxidative conditions, the WS_2 phase appears to be more stable, which broadens the applicability of this lubricating agent [7]. In the case of temperature unstable phases such as MoS_2, the synthesis of sliding layers may be accomplished as described in Deng, Song, Zhang [8]. WC+8%Co-based cutting plates with an aperture (made using the micro-EDM method) and a lubricating agent placed within this were characterised by a lower coefficient of friction and the forces generated during cutting of hardened steel were lower than for plates without the addition of lubricating agent. A similar method of preparing the contact surface and, in the case of friction, production of a lubricating film is presented in Voevodin, Zabinski [9]. Apertures are made with a laser in a TiCN layer on a nickel alloy (Inconel 718). Used as lubricating agents were: MoS_2, and MoS_2/graphit/Sb_2O_3 powders. Another method of producing an MoS_2 sliding layer is presented in Smorygo, Voronin, Bertrand, Smurov [10]. The MoS_2 layer was produced on high-speed steel, in two stages. In the first stage, MoO_3 powder is put on the steel suraface and heated at a temperature of 550-750°C for 8 h. Then the steel with a layer of MoO_3 is subject to heating in MoS_2 powder (temperature 480-525°C; time 5-7 h; vacuum 10^{-1} Pa). Tools with a sliding layer thus produced were characterised by lower wear and higher durability as compared to uncoated tools.

Taking into account temperature resistance (resistance to an oxidative environment), lubricating agents based on graphite, molybdenum disulphide or tungsten disulphide can be used at fairly low temperatures. In the case of high-speed machining, solid sliding agents should be characterised by a low coefficient of friction and resistance to oxidation at temperatures exceedomg 800°C. In such a case, layers are used composed of high- and low-temperature phases, e.g. CaF_2 or WS_2 which, as a result of the reaction to temperature and friction become more temperature stable phases. In accordance with this mechanism, phases can be formed which are characterised by good tribological properties at high temperatures, for example: $PbMoO_2$ and $ZnWO_4$ (MoS_2+PbO→$PbMoO_4$; WS_2+ZnO→$ZnWO_4$) [11]. On the basis of the results of mechanical and tribological studies presented in Deng, Can, Sun [12] it may be stated that the content of sliding agents in tool ceramics should not exceed 10%. In this work, the effect is analysed of sliding agents CaF_2, MoS_2 and cBN on the properties of Al_2O_3/TiC ceramics. Among the above-mentioned sliding agents, the fluorite CaF_2 is characterised by the best tribological properties and highest stability, including during sintering. Similar results have been presented in other works [13, 14, 15]. On the basis of tribological studies, it is ascertained that the addition of CaF_2 reduces the coefficient of friction of Al_2O_3/TiC-based ceramics from a value of 0.5 to 0.3. It should be noted, however, that the addition of a sliding agent results in a significant reduction in hardness and other mechanical properties of tool ceramics, as evidenced by the results presented in Jin, Kato, Umehara [16]. Materials Al_2O_3-CaF_2, Al_2O_3-Ag-CaF_2 and Al_2O_3-Ag-CaF_2 were formed by HIP sintering at temperatures of 1150-1250°C. The materials obtained were, however, characterised by a fairly low hardness, at 30-50% the hardness of Al_2O_3 ceramics. Whilst the addition of a sliding agent to Al_2O_3-based ceramics precludes their use in cutting tools, the possibility of reducing the CaF_2 sliding phase content by the addition of a metallic (Ag) phase appears interesting. It is therefore necessary to determine the optimal level of sliding agent content.

Among those materials characterised by a higher temperature resistance than graphite or MoS_2, worthy of mention are: $CaSO_4$, $BaSO_4$ and $SrSO_4$. As stated in other works [17, 18] sulphate-based sliding phases are characterised by a coefficient of friction of 0.15, even at temperatures of around 600°C. $PbMoO_4$, $ZnMoO_4$ and $ZnWO_4$ layers have similar properties (coefficient of friction, wear resistance, high-temperature wear resistance). At low temperatures, sulphate-based sliding layers and those containing MoO_4^{2-} or WO_4^{2-} ions are characterised by a higher coefficient of friction and are rapidly damaged. This is confirmed by the results of tribological studies presented in John, Zabinski [18]. The high temperature resistance and good tribological properties of $SrSO_4$ layers is confirmed by the results presented in Murakami, Umeda, Sasaki, Ouyang [19]. Al_2O_3-based ceramics and Si_3N_4 were coated by chemical reaction with a layer of $SrSO_4$. Al_2O_3-ZrO_2-Y_2O_3 ceramics coated with an $SrSO_4$ layer are characterised by a low coefficient of friction and good abrasion resistance up to 800°C in an oxidative atmosphere. In the case of Si_3N_4 ceramics coated with an $SrSO_4$ layer, a reduction was ascertained in wear rate due to friction at a temperature of approximately 400°C. A promising material for use in tool ceramics as a solid lubricating agent is $BaCrO_4$. This phase is characterised by a low and constant coefficient of friction over a wide temperature range, up to approximately 800°C. $ZrO_2(Y_2O_3)$-$BaCrO_4$ ceramics produced by SPS (Spark Plasma sintering) are characterised by a higher resistance to abrasive wear, in comparison to $ZrO_2(Y_2O_3)$-based ceramics [20]. Furthermore, it is ascertained that the addition of a $BaCrO_4$ sliding phase results in a change in the mechanism of wear from brittle fracture to plastic deformation.

The aim of this study is to determine the mechanical and tribological properties of Al_2O_3-Ti(C,N)-based ceramics with added sliding phases, produced by free sintering.

2. Experimental procedure

Al_2O_3-Ti(C/N)-based tool ceramics with the addition of various lubricating substances were subjected to investigation. The base material was composed of the following powders: 68 mass.% Al_2O_3 (A16SG, ALCOA, 0.7μm); 2 mass.% ZrO_2 (FLUKA, <0.5 μm); 30 mass.% Ti(C,N) (H.C. STARCK, ~1 μm). To the base material was added 5 mass.% powder: MoS_2 (Mo-801, AEE, 1-5 μm); Mo (GOODFELOW, 2 μm); $BaZrO_3$ (ALDRICH, <10 μm); $CaSO_4$ (POCH); $Al_2[Si_4O_{10}](OH)_2$ (<10 μm). The designation and chemical composition of individual materials is presented in Table 1.

Individual mixtures were prepared in a colloid mill and then granulated using a sieve with a mesh size of 0.9 mm. Pressing was carried out in a uniaxial steel die at a pressure of 110 MPa and cold isostatic pressing at a pressure of 250 MPa. After drying, with programmed temperature increases up to 210°C, compacts were sintered in a BALZERS vacuum furnace. Materials were sintered at a temperature of 1750°C under vacuum conditions of $5x10^{-4}$ Pa. After sintering, materials were subjected to a study of physical and mechanical properties. Apparent density ρ_p, was measured using the hydrostatic method. Young's modulus measurements of sintered samples were also taken, using the ultrasonic method of measuring transition speed of transverse and longitudinal waves, using a Panametrics Epoch III flaw detector. Hardness was determined by the Vickers method at a load of 9807 mN using a digital hardness tester (Future Tech. Corp. FM-7). For these studies, metallographic specimens were prepared using Struers machines and polishing agents. The sintered materials were subject to observation with a JEOL JSM-60LV scanning electron microscope. Chemical composition analysis in micro-areas of individual samples was performed using an EDS-INCA ENERGY 350 X-ray microanalyser. Tribological studies were performed using a UMT-T2 universal testing machine. Analysis of tribological properties of individual materials was performed at a temperature of 22°C.

3. Results and discussion

Results of measurements taken of density, Young's modulus and Vickers hardness are contained in Table 1. Irrespective of the type of sliding phase, values of relative density, hardness and Young's modulus of individual materials were lower than those recorded for the base material Al_2O_3-Ti(C,N). Such a significant reduction in values (particularly relative density) for materials with added sliding phases may result from their partial decomposition at sintering temperature and the emergence of gas phases as decomposition products. Figure 1 presents the microstructure of Sample 2 (with added MoS_2); chemical composition analysis in micro-areas is contained in Table 2. This material was characterised by a relative density of 89% and a Vickers hardness of over 1300 HV1. Figure 2 presents the microstructure of Sample 3 with added $BaZrO_3$ chemical composition analysis in micro-areas is contained in Table 3. For this material were recorded the lowest values of relative density (85%), Young's modulus (273 MPa) and hardness (966 HV1). This material was, furthermore, characterised by a high heterogeneity, as shown by clusters of ZrO_2 phases (light area). The microstructure and chemical composition analysis in micro-areas for Sample 4 (with added $CaSO_4$) are presented in Fig. 3 and Table 4. This material was characterised (similarly to Sample 2) by a high heterogeneity of the carbonitride phase Ti(C,N). For this material was recorded the lowest value of relative density, a Vickers hardness of over 1200 HV1 and a Young's modulus of 311 MPa. Similar values of relative density and hardness (HV1) were recorded for Sample 5 to which, besides $CaSO_4$ was also added molybdenum. This material was characterised by a higher Young's modulus value (343 MPa). The microstructure and results of EDS analysis for this material are presented in Fig. 4 and Table 5. In the case of the material with added $Al_2[Si_4O_{10}](OH)_2$, the relative density value was 88.8%, Young's modulus 288 MPa and hardness 1221 HV1. The microstructure of this material is presented in Fig. 5. Chemical composition analysis of Sample 6 at points 2 and 7 and micro-area 8 (Table 6) reveals the emergence of a TiC carbide.

Table 1.
Results of measurements of density, Young's modulus and hardness for ceramics based on Al_2O_3+30wt.%Ti(C,N)

No.	Type of material	Sintering temp. T	Relative density	Elastic modulus E	Hardness HV1	
		[°C]	[%]	[GPa]	-	s
1	Al_2O_3-Ti(C,N)	1700	96.3	385	1790	65
2	Al_2O_3-Ti(C,N) + MoS_2	1750	89.2	304	1373	93
3	Al_2O_3-Ti(C,N) + $BaZrO_3$	1750	85.3	273	966	42
4	Al_2O_3-Ti(C,N) + $CaSO_4$	1750	92.3	311	1256	140
5	Al_2O_3-Ti(C,N) + Mo + $CaSO_4$	1750	91.4	343	1264	121
6	Al_2O_3-Ti(C,N) + $Al_2[Si_4O_{10}](OH)_2$	1750	88.8	288	1221	127

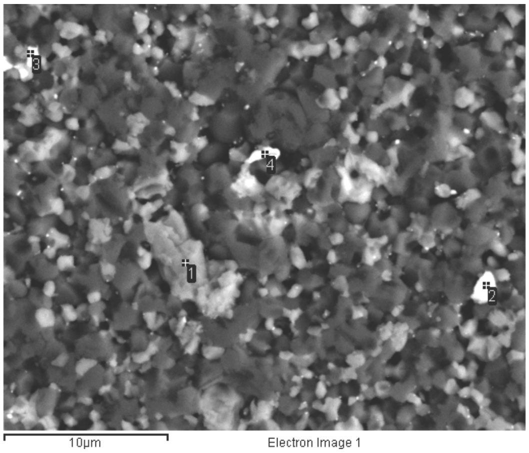

Fig. 1. SEM microstructure of sample Al_2O_3-Ti(C,N)+MoS_2

Table 2.
Chemical composition of sample Al_2O_3-Ti(C,N)+MoS_2

Element [mass.%]	Area 1	2	3	4
C	10.41	16.88	15.72	12.40
N	11.54	-	-	-
O	-	12.02	47.23	42.11
Al	0.35	1.73	29.47	28.43
Ti	77.05	2.58	4.20	9.34
Zr	0.65	1.51	-	-
Mo	-	65.27	3.38	7.73

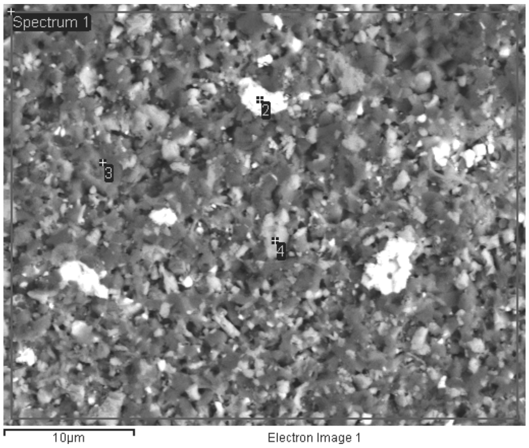

Fig. 2. SEM microstructure of sample Al_2O_3-Ti(C,N)+$BaZrO_3$

Table 3.
Chemical composition of sample Al_2O_3-Ti(C,N)+$BaZrO_3$

Element [mass.%]	Area 1	2	3	4
C	4.59	12.56	5.89	5.78
N	-	-	-	14.56
O	47.88	26.02	50.57	13.94
Al	26.70	6.39	38.76	3.62
Ti	15.13	1.49	4.20	62.10
Zr	3.88	53.56	0.58	-
Ba	1.82	-	-	-

Table 4.
Chemical composition of sample Al_2O_3-Ti(C,N)+$CaSO_4$

Element [mass.%]	Area 1	2	3	4
C	9.40	12.63	15.52	6.64
N		4.19	-	-
O	47.27	9.69	43.49	48.56
Al	23.94	0.16	39.75	38.56
S	0.42	-	-	-
Ca	1.08	0.68	0.31	4.07
Ti	16.57	72.65	0.93	1.22
Zr	1.31	-	-	0.95

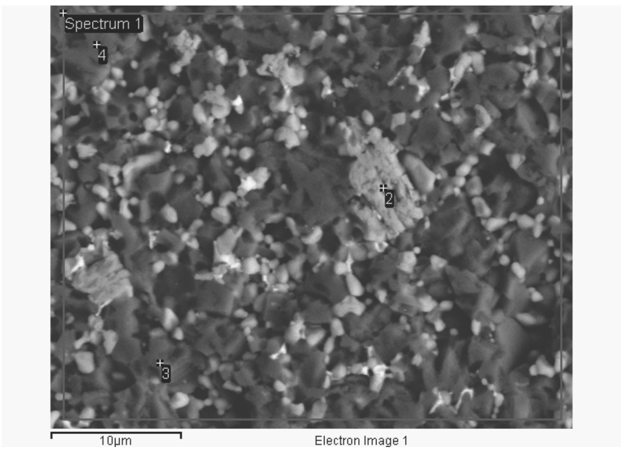

Fig. 3. SEM microstructure of sample Al_2O_3-Ti(C,N)+$CaSO_4$

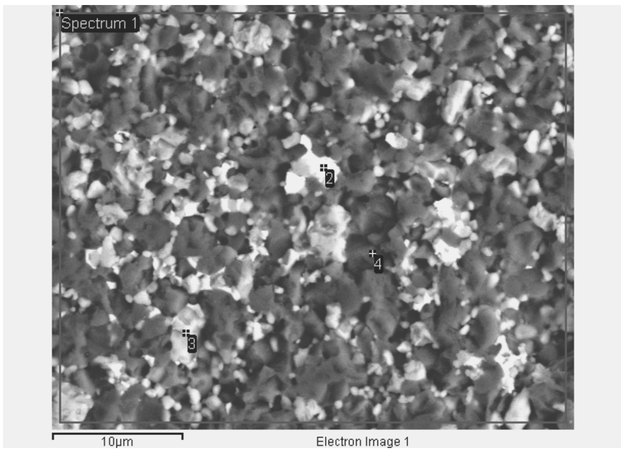

Fig. 4. SEM microstructure of sample Al_2O_3-Ti(C,N)+Mo+$CaSo_4$

Table 5.
Chemical composition of sample Al_2O_3-Ti(C,N)+Mo+$CaSO_4$

Element [mass.%]	Area 1	2	3	4
C	7.09	13.24	9.77	8.95
N	-	6.89	4.46	-
O	48.71	9.04	17.61	49.93
Al	25.11	0.75	1.34	40.10
Ca/S	0.68/0.47	-	-	-
Ti	16.61	7.93	64.27	1.02
Mo	1.33	62.15	2.56	-

Table 6.
Chemical composition of sample Al_2O_3-Ti(C,N) with $Al_2[Si_4O_{10}](OH)_2$

Area	Element in mass. % C	N	O	Al.	Si	Ti
1	9.49	-	48.72	26.51	0.61	14.67
2	69.20	-	10.05	6.66	0.11	13.98
3	29.83	-	39.57	21.90	-	8.70
4	7.44	22.71	-	0.37	-	69.48
5	-	-	51.91	47.78	-	0.31
6	-	-	51.42	46.94	0.20	1.43
7	9.13	-	20.11	20.18	-	50.57
8	25.94	-	43.57	18.58	0.27	11.64

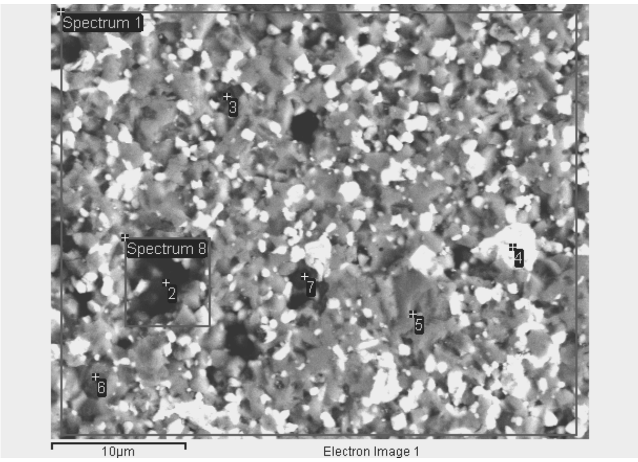

Fig. 5. SEM microstructure of sample Al_2O_3-Ti(C,N) with $Al_2[Si_4O_{10}](OH)_2$

In tribological studies, hardened steel balls of diameter 3.175 mm were used as the counter-body. Parameters of tribological analysis: ball load 10 N; velocity v=0.1 m/s; distance s=100 m; test time 1000 s. Roughness of samples for tribological studies: R_a=0.8 µm.

As a result of these tribological studies (Table 7), the coefficient of friction value for the base material was determined to be 0.51. For the other materials, the coefficient values were in the range 0.28 to 0.41, the lowest value being for the material with the addition of $BaZrO_3$. The addition of MoS_2, $CaSO_4$ and Mo+$CaSO_4$ to the base material allowed the following respective coefficient of friction values to be achieved: 0.34; 0.38; 0.36.

In the case of the base material with the addition of $BaZrO_3$, the lowest friction resistance was achieved, the mechanism for this process being not yet sufficiently known. In contrast to the other sliding additives, $BaZrO_3$ is characterised by a high-symmetry crystal structure and its content by volume did not exceed 4%. An explanation for this may be a change in the extra-complex cation (complex symmetry change) and the emergence of derived hettotypes or hettotypes with an altered crystal structure. The contents by volume of the other sliding additives were respectively: MoS_2 - 4.6%; $CaSO_4$ - 8.4%; $MgCO_3$ - 3.9%; $Al_2[Si_4O_{10}](OH)_2$ - 7.9%; Mo - 2.2%; Mo+$CaSO_4$ - 1.1 + 4.4%. Further, the majority of these additives are characterised by a layered structure or possess cleavage planes, which should reduce friction resistance and allow the formation of a lubricating film on the surface of the sample. The higher friction resistance of these materials demonstrates that, as a result of sintering at high temperatures, portions of the sliding additives underwent decomposition and, in consequence, their content by volume was reduced.

4. Conclusions

Presented in this study are the results of mechanical and tribological studies of the Al_2O_3-Ti(C,N)-based ceramics with additives to reduce friction resistance. Irrespective of the type of additive and its content by volume, it was possible to reduce the coefficient of friction to between 55% and 80% of the coefficient of friction of the base material. Given, however, that, for some additives, a significant reduction in Vickers hardness and Young's modulus was recorded, further studies should focus on the limiting of the content by volume of the additives and simultaneously the increasing of their degree of dispersion.

Table 7.
Results of tribological analysis for ceramics based on Al_2O_3+30 mass.%Ti(C,N)

Nr	Type of material	Friction radius	Velocity		Number of cycles	Distance s	Coefficient of friction	
		[mm]	[m/s]	[rpm]	-	[m]	COF	s
1	Al_2O_3-Ti(C,N)	3	0.100	318.3	5305.2	100	0.51	0.06
2	Al_2O_3-Ti(C,N) + MoS_2	1	0.100	954.9	15915.5	100	0.34	0.02
3	Al_2O_3-Ti(C,N) + $BaZrO_3$	4	0.100	238.7	3978.9	100	0.28	0.06
4	Al_2O_3-Ti(C,N) + $CaSO_4$	4	0.100	238.7	3978.9	100	0.38	0.04
5	Al_2O_3-Ti(C,N) + Mo + $CaSO_4$	4	0.100	238.7	3978.9	100	0.36	0.04
6	Al_2O_3-Ti(C,N) + $Al_2[Si_4O_{10}](OH)_2$	4	0.100	238.7	3978.9	100	0.37	0.04

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