
Characterization of Al₂O₃/Al/Al₂O₃ joints with Ti+Cr interlayer

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Abstract: Joints of Al₂O₃/Al/Al₂O₃ are formed by liquid – state bonding of alumina substrates covered with thin Ti+Cr film of 380 nm thickness using an Al interlayer of 30 μm at 973 K under a vacuum of 0.2 mPa applied for 5 minutes and at a pressure of 0.01 MPa. The bond strength of the joints is examined by a four – point bend testing at room temperature, coupled with optical, scanning and transmission electron microscopy. Results show that introduction of the thin Ti+Cr film layer: (i) improves plasticity in the metal layer of Al₂O₃/Al/Al₂O₃ joints, (ii) has positive effect on structure transformation in the interface and leads to fabrication of reliable metal-ceramic joints. Information obtained from the wetting characteristics of liquid Al on alumina substrate coated by thin Ti+Cr film indicates that the contact angle can be reduced to even less than 60°. Microstructural investigations of the interface of Al₂O₃/Ti+Cr/Al/Ti+Cr/Al₂O₃ joints carried out by SEM and TEM techniques indicated that the precipitates of intermetallic phases rich in both Ti and Cr as well as Al₂O₃ were formed at the Al/Al₂O₃ interface, which influenced strengthening of these joints. Hence a conclusion can be drawn that the interface structure influences the mechanical durability increase in Al/Al₂O₃ joints.

Keywords: Metal–Ceramic Joining, Liquid State Bonding, Bond Strength, Fracture Interface

1. Introduction

Aluminum/alumina systems have an increasing importance in, for example, joining of composites or coatings technology. In many of these cases, the formation of a strong aluminum/alumina interface is required. The reliability of these systems depends on the interfacial properties of metal and alumina, e.g. adhesion properties and thermal stability. Therefore a better understanding of the reaction at these interfaces and of the thermomechanical properties of the metal and its ability to accommodate residual stresses, induced during the process of joining is required.

Liquid state bonding is a reliable and cost-effective process for production of metal-ceramics joints. The main problem encountered in this technique of joining is how to achieve optimal wetting of the ceramic substrate by the liquid metal. In order to enhance the wettability, different solutions are used, e.g., applying active elements to the metal and ceramic pre-metallization [1-3]. Some active

elements such as titanium have been studied both as active metal additions and as a coating on ceramic to improve the wettability of ceramic by liquid metal. However, the good wetting characteristics offered by Ti are counteracted by undesirable intense reactions at the metal-ceramic interface such as the reduction of alumina and formation of titanium oxide or of brittle intermetallic phases [4]. Thus, the preferred solution seems to be the use of additive elements which can improve wettability without extensive chemical interaction in the area of bond interface. Studies of chromium have shown that this element increases the bond strength of metal/Al₂O₃ joints without any significant chemical interaction in the area of bond interface [5,6]. Therefore extensive research is being performed on the wetting behavior of liquid Al on alumina substrate and on the liquid-state joining of both materials. Modification of the ceramic surface with thin complex metallic film of Ti+Cr was proposed for designing high-strength metal-ceramic joints.

The aim of this work is to explain the wetting

characteristics of Al on alumina substrate covered with thin Ti+Cr layer, and the effect this layer may have on the structure and mechanical properties of an aluminum/alumina interface. Joints of $\text{Al}_2\text{O}_3/\text{Ti}+\text{Cr}/\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ were fabricated by liquid - state bonding to investigate the effect of modification of the surface of ceramics with a thin Ti+Cr film on metal-ceramic joining.

2. Experimental Procedure

The following materials were used in the investigations: pure aluminum (99.9999 wt. % for wetting experiment and 99.99 wt.% for liquid state bonding) and 98% pure polycrystalline alumina, fabricated at the Faculty of Materials Science and Ceramics, University of Science and Technology in Cracow, by sintering at the temperature of 1923 K for 2 h, powders of the following starting composition: 99.9085 % $\alpha\text{-Al}_2\text{O}_3$, 0.009 % CaO, 0.053 % SiO_2 , 0.0029 % MgO, 0.023 % Fe_2O_3 and 0.0036 % Na_2O . The alumina was characterized by the following physical and mechanical properties: porosity - almost 0%, density - 3.8 g/cm³, Young's modulus - 300 GPa, Poisson ratio - 0.24.

The effect of modification with a thin film of Ti+Cr on the wetting characteristics of liquid Al on Al_2O_3 has been studied by a sessile drop method. The wettability experiment was carried out in the temperature range of 1023 - 1123 K for 30 minutes in a dynamic vacuum of 0.2 mPa, heating the samples with furnace from ambient temperature to 1023 and 1123 K. After wettability examinations, the Al/ Al_2O_3 couple was cooled at a rate of 10 K/min to reduce the effect of thermal stresses. Metal specimens weighing 0.20 g were used in the tests. The samples of Al_2O_3 substrates of $\phi 17 \times 5$ mm dimensions were cut out from the sintered blocks of $\phi 17 \times 40$ mm dimensions using a diamond saw, and were polished with diamond paste of 1 μm grain size to the surface roughness of about 0.3 μm . As a next step, onto the alumina substrates, the film of Ti+Cr (380 nm thick) was deposited separately by PVD method. The film was deposited by rf sputtering of a mosaic Ti + Cr target in argon gas atmosphere onto different substrates such as Corning glasses and alumina, depending on the requirements imposed by further applications i.e., for determination of the film thickness and composition. The film thickness was measured mechanically by means of the Taly Step profilometer. The thin film composition was determined from an X - ray diffraction pattern (XRD). The weight ratio of Ti/(Ti + Cr) was found to be equal to 0,580 (58,0 wt. %).

On joint specimens examined by the sessile drop method, the bond strength was next tested by a shear method (modified push - off technique). In this technique, the solidified sessile drop specimens are carefully bisected perpendicular to the drop-substrate interface at the mid-plane of the contact circle by means of an Accutom 50 automatic precision cut-off machine and the shear test is done on one half of the specimen. Additionally, the other

half of the same bisected droplet may be used to carry out detailed structure and chemistry characterization of interfaces formed under conditions identical to the corresponding wettability test, see: Ksiazek et al. [7]. The bond strengths were determined using an INSTRON 1115 machine with a special holder, and with automatic recording of the applied load versus the corresponding displacement up to failure under constant displacement rate of 1 mm min⁻¹. The shear strength was calculated dividing the applied load by the real shear area measured under x 10 magnification using an AMSLER magnifying system. For calculation of the real shear area (assuming that this area is part of a circle of radius R) considered cases, where radius of the real shear area is equal to, less or greater than the way of shear. Due to the fact, that shear stress at the interface is not simple shear and it always contains the component of tensile stress originated from the bending moment, which cannot be neglected, the shear test is not recommended for the common evaluation method.

Liquid state bonding was also conducted in a vacuum furnace used to make sandwich specimens with 30 μm thick layers of pure aluminum (99.99%) bonded between 98% pure polycrystalline Al_2O_3 . The alumina blocks (14x14x20 mm dimensions) of 3.8 g/cm³ (almost 0% porosity) were produced by the same technique and from the same powders as for the wettability experiment. The surfaces to be joined were lapped flat to a 1 μm finish, ultrasonically cleaned in acetone, and finally a 380 nm thin Ti+Cr layer was deposited on the surface by Physical Vapor Deposition (PVD). The Al foil was mechanically and ultrasonically cleaned in acetone, for 10 min rinsed in alcohol and then dried with warm air directly before bonding. The Al foils were placed between two Al_2O_3 blocks and this assembly was placed in a vacuum chamber under a load applied by placing one of the Al_2O_3 blocks on the top of the assembly (an applied pressure of 0.01 MPa). Bonding was carried out in a vacuum of 0.2 mPa at a temperature of 973 K for 5 minutes before cooling at a rate of 30 K min⁻¹ to room temperature.

Samples for bend testing of dimensions 3 mm x 3 mm x 36 mm were cut out from the internal part of microcrack-free joints using a low - speed diamond saw, with the joint perpendicular to the long axis of the specimen. The lines on specimen surfaces in contact with the applied load were polished down with 1 μm diamond paste. Beams were tested at room temperature using four-point bending with an outer span of 25 mm and an inner load application point span of 12.5 mm. The specimens were tested using an INSTRON 1115 machine with automatic recording of the applied load versus the corresponding displacement up to failure under a constant load displacement rate of 1 mm/min. The reported results are averages of between three and five tests run for each condition.

The structure was examined on specimens after the wettability test (metal drop - ceramic substrate couple) and on sandwich specimens after joining, using optical microscopy, scanning electron microscopy and

transmission electron microscopy. The examinations of structure were restricted to the areas adjacent to metal–ceramic interface. The bond structure morphologies present in the interface were determined by scanning microscopy on a STEREOSCAN 420 scanning microscope with analysis of the metal-ceramic interface chemical composition done on an EDS LINK ISIS 300 X-ray microanalyzer. The application of X-ray microanalysis enabled the linear, local and surface distribution analyses to be carried out in the examined microregions of metal-ceramic interface. Detailed structural examinations of the joints were also carried out on a JOEL 2010 ARP transmission electron microscope equipped with Energy Dispersive X-ray spectrometer (EDS). The TEM examinations of the structure of metal-ceramic joints were made on thin foils prepared in a Gatan PIPS691V3.1 ion thinner by standard methods, i.e. by cutting out 3 mm diameter discs, thinning by dimpler, and ion polishing [8].

In the case of samples derived from joining technique (sandwich sample) structural investigations were performed on fractured joint specimens, cross sectioned in a plane inclined at about 25 degrees to the joint surface (to enable enlargement of the joint area available for examinations; it is noted that this technique lowers somewhat the spatial resolution of EDS composition line scans).

3. Results and Discussion

The effect of modification with a Ti+Cr thin film on the wetting characteristics of liquid Al on Al₂O₃ has been studied using a sessile drop method. Figure 1 shows the wetting kinetics of Al/Al₂O₃ and Al/Ti+Cr/Al₂O₃ systems at temperatures of 1023 K and 1123 K during 30 minutes of contact. Within the temperature range of 1023 K – 1123 K, the contact angle in base Al/Al₂O₃ system is larger than 90°. The application of Ti+Cr thin film has no effect on reducing the contact angle of liquid Al on Al₂O₃ at the temperature of 1023 K. However, at 1123 K, the Ti+Cr coating reduces the contact angle of liquid Al on Al₂O₃ ($\Theta=55^\circ$), which proves good wettability and intense chemical reaction proceeding at that temperature.

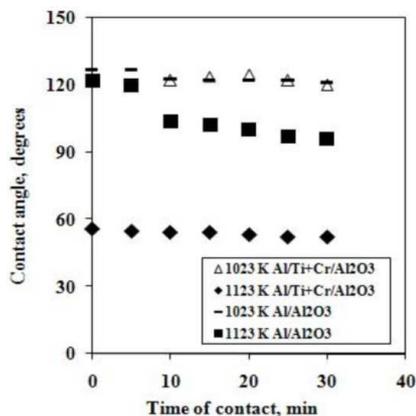


Figure 1. Wetting kinetics of Al/Al₂O₃ and Al/Ti+Cr/Al₂O₃ systems at temperatures of 1023 K and 1123 K.

Moreover, it should be noted that the contact angle gives information on the magnitude of the interfacial bonding. The contact angle of the Al/Ti+Cr/Al₂O₃ is influenced by both the content of Ti and Cr and a ratio of Ti to Cr. The concentration of Ti in liquid Al is reduced by the additions of Cr, which decrease the solubility but increase its activity and lead to an improvement of wettability [9].

For the explanation of the mechanism controlling the wetting process in Al/Ti+Cr/Al₂O₃ system, the structural examinations carried out in the region of Al/Ti+Cr/Al₂O₃ bond by the method of optical microscopy, combined with SEM and TEM, are shown in Figs. 2, 3. Observations under a magnification of 500x revealed a rather smooth interface and in the area of bond interface, locally, the presence of a new phase of fine precipitates, marked in the microstructural images with arrows (Fig. 2a). SEM observations (Fig. 2b, c) have proved the formation of new phases, i.e., a phase containing mainly Ti, and also a phase containing oxygen. Local analysis of the chemical composition of the above mentioned precipitates has indicated that these are the precipitates of intermetallic phases rich in Ti as well as Al₂O₃ (e.g., items 1, 2 and 3 in Fig. 2b, respectively). The chemical composition in the interface region also shows maps of the distribution of the elements like aluminum, titanium, chromium, silicon and oxygen (Fig. 2c).

This statement is consistent with the results of analysis of the Al–Ti–Cr phase equilibrium diagrams based on the reaction at 1073 K [10]. According to a phase diagram several intermetallic phases such as Cr(Cr₂Al), Al₁₇Cr₉, Al₃Ti, Al₇Ti₃, TiAl, TiAlCr, and Laves phase, (Al,Cr)₃Ti with L12 structure may be formed. Here it is worth noting that, because of difficulties in correct estimation of the chemical composition of an Al drop after its interaction with the Ti+Cr film coating, it has been assumed that the thin film layer (about 380 nm thick) results in the actual Ti+Cr content lower than 0.5 wt. %. The structural analysis of the Al/Ti+Cr/Al₂O₃ bond interfaces produced in the temperature range of 1023 - 1123 K indicates that the wettability effect has been accompanied by intense chemical interaction between the Ti+Cr film coated substrate and liquid aluminum. The mechanism of this interaction is probably due to dissolution of Ti+Cr coating in liquid aluminum. The result is diffusion and, as a consequence of this diffusion, a reaction between aluminum and the metallic coating, resulting in the formation of new intermetallic phase rich in Ti. The energy dispersive spectrometry (EDS) analysis in Fig. 2 indicates Al and oxygen within the interface area. At this point of the investigations, a hypothesis can be put forward that Al₂O₃ oxide nucleates within the interface area in the presence of oxygen originating from the dissolved oxide coating deposited on Al drop and from the surface of the ceramic material. Oxygen originating from the oxide coating present on Al drop and/or from the surface of the ceramic material is entrapped in the area of the interface, and its presence may result in the achievement of local

thermodynamic equilibrium, which promotes the formation of Al_2O_3 . Due to these changes, oxygen is sucked off, and its absence in the interface area arrests the reaction, resulting in the formation and growth of oxides. This is why the composition and morphology of phases formed at the $\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ bond interface depend on temperature, pressure, atmosphere, contact time, and partial oxygen pressure $p(\text{O}_2)$ [11]. Moreover should be taken into consideration in the analysis and interface formation evaporation of Cr in the test chamber.

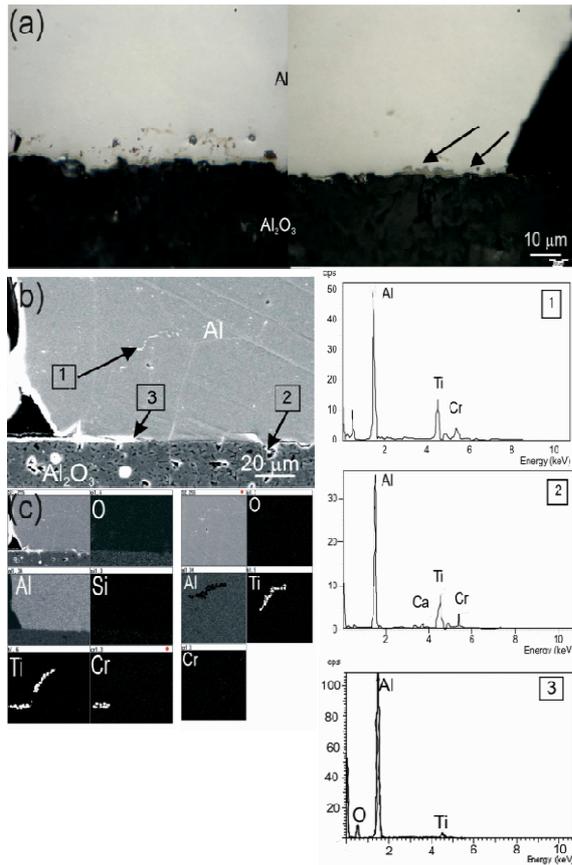


Figure 2. Images of cross section of the $\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ sample after wetting experiment at 1023 K: (a) optical, (b) SEM with corresponding EDS spectra taken from the marked points: 1, 2 – the Ti-rich precipitates at the interface, 3 – Al_2O_3 precipitates in the metal penetrated region in the substrate – side interface (c) Al, O, Si, Ti, Cr X-ray maps taken from the region of the interface and Al, O, Ti X-ray maps taken from the drop-side interface.

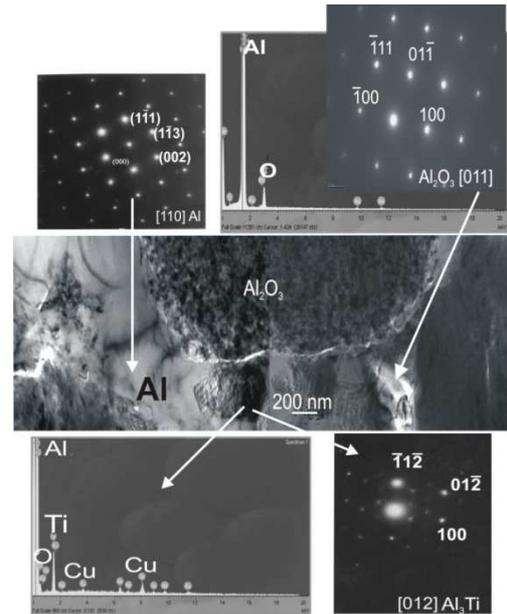


Figure 3. TEM analysis of the $\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ interface with corresponding EDS spectra and selected area diffraction patterns from the phase rich in Al, the Al_3Ti precipitates and Al_2O_3 phase.

The last statement was confirmed experimentally by TEM studies of the $\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ system interface, and the presence of Ti-rich phases and isolated separate particles of Al_2O_3 in the region of the interface (Fig. 3). The diffraction pattern taken for a selected area in this region and EDS spectrum confirmed the presence of the precipitates with a chemical composition similar to Al_3Ti phase and of an Al_2O_3 phase. The results of a shear test, carried out by modified *push-off* technique on an $\text{Al}/\text{Al}_2\text{O}_3$ and $\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ system, are compared in Figure 4 as a shear stress - displacement relationship at the wettability test temperatures of 1023 and 1123 K. Compared with basic system, the values of the maximum shear stresses are higher for the $\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ bonds at the wetting test temperatures equal to 1023 K and 1123 K. Maximum values of the shear stresses obtained in tests carried out on the $\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ system at the temperatures of 1023 K and 1123 K amount to 40 and 46 MPa, respectively. The reason is most probably the presence of fine precipitates of phases rich in Ti, having more prominent effect on hardening of this zone, as well as the presence of Al_2O_3 crystals that adhere more strongly to the interface, thus improving the general adhesive behavior of a metal-ceramic system.

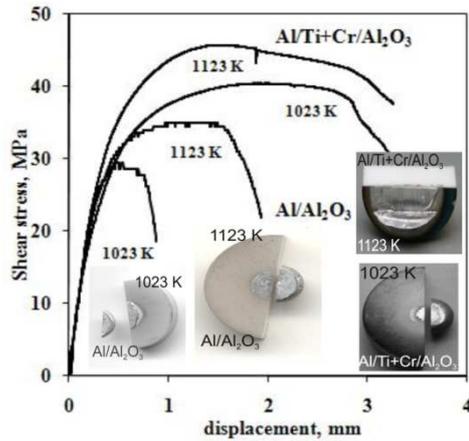


Figure 4. Shear stress as a function of displacement for the $\text{Al}/\text{Al}_2\text{O}_3$ and $\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ sessile drop samples obtained after wettability test at 1023 K and 1123 K.

This suggested that the changes in interlayer chemistry could improve the wetting characteristic of the interface and thus lead to strength improvement. Despite the lack of wettability in the system with coating up to 1023 K, chemical interaction in the interface area is so intense that the area is strengthened.

Figure 5 compares the results of bending tests performed on $\text{Al}_2\text{O}_3/\text{Ti}+\text{Cr}/\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3/\text{Al}/\text{Al}_2\text{O}_3$ joints with Al interlayer of 30 μm thickness produced at 973 K with a pressure of 0.01 MPa. Specimens with coatings displayed an average breaking stress of 188 MPa with a standard deviation of ≈ 15 MPa, the deflection value corresponding to the maximum fracture stress was approximately 172 μm , while specimens without the coatings failed at a breaking stress higher by 100 MPa, again with a standard deviation of about 15 MPa and at a much lower deflection of 113 μm . An important element in these investigations was the location of the place of joint destruction. In $\text{Al}_2\text{O}_3/\text{Al}/\text{Al}_2\text{O}_3$ joints, characterized by high bond strength, failure occurred by brittle cracking in the alumina near the interface (Fig. 5). On the other hand, in $\text{Al}_2\text{O}_3/\text{Ti}+\text{Cr}/\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ joints, failure occurred in the Al layer, as evidenced by a large non-linear region present in the stress-deflection curve after the bend stress has reached its maximum value, as compared to these specimens which failed in the ceramic. Thus, plasticity in the metal layer in $\text{Al}_2\text{O}_3/\text{Ti}+\text{Cr}/\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ joint is the valid parameter determining the integrity and lifetime of ceramic/metal/ceramic layered structure. The “strengthened” region of the interface by thin film of Ti+Cr reduces the residual stress by elastic, plastic and creep deformation. In addition thin film of Ti+Cr decreases of thermal expansion coefficient mismatch ($\alpha_{\text{Al}}=23.8 \times 10^{-6} \text{ K}^{-1}$, $\alpha_{\text{Al}_2\text{O}_3}=8 \times 10^{-6} \text{ K}^{-1}$, $\alpha_{\text{Ti}}=8,64 \times 10^{-6} \text{ K}^{-1}$, $\alpha_{\text{Cr}}=6.2 \times 10^{-6} \text{ K}^{-1}$) which contributes to improving of reliable $\text{Al}_2\text{O}_3/\text{Al}/\text{Al}_2\text{O}_3$ joints (lack of micro-cracks at the interface after cooling). Therefore such region of the interface provides an effective way for the compensation of residual stresses and obtaining reliable and strong joints.

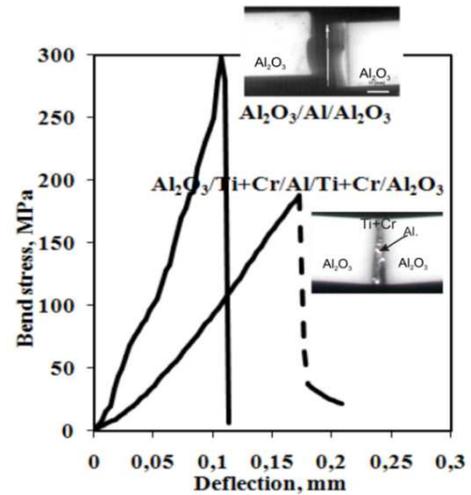


Figure 5. Bend test curves recorded for $\text{Al}_2\text{O}_3/\text{Al}/\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3/\text{Ti}+\text{Cr}/\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ joints.

The results of structure examinations carried out by optical microscopy in the region of the $\text{Al}_2\text{O}_3/\text{Ti}+\text{Cr}/\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ joint show the presence of fine precipitates of the new phase distributed in the form of chains along the interface (Fig. 6a).

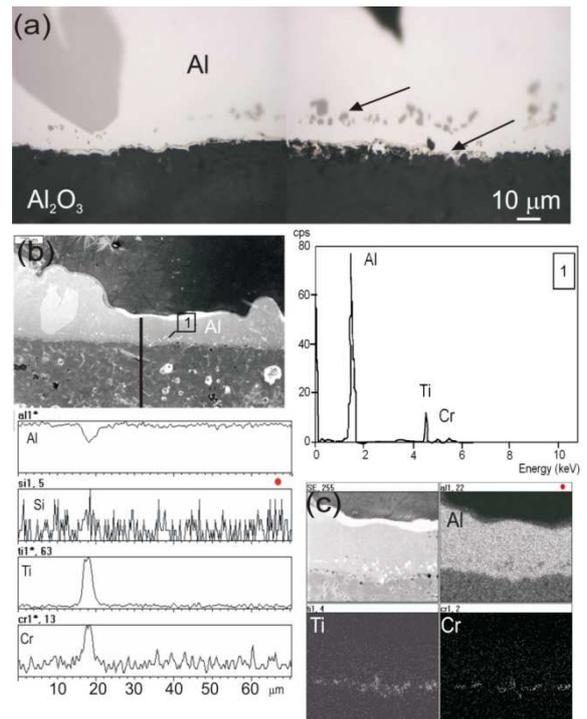


Figure 6. Images of cross section of the $\text{Al}_2\text{O}_3/\text{Ti}+\text{Cr}/\text{Al}/\text{Ti}+\text{Cr}/\text{Al}_2\text{O}_3$ sandwich sample: (a) optical, (b) SEM with corresponding EDS spectrum taken from the marked point 1 - the complex Ti, Cr rich precipitates at the interface and distribution of Al, Si, Ti and Cr along lack line marked in (b), (c) map of distribution of concentrations of Al, Ti and Cr taken from the region of the interface.

Detailed structural examinations carried out by scanning microscopy (Fig. 6b, c) lead to a conclusion that the complex of titanium and chromium – containing phase, as identified from the measurements of chemical composition

(Fig. 6b: item 1 and distribution of concentrations of Al, Ti and Cr along black line). The presence of the complex phase rich in both Ti and Cr is further confirmed qualitatively by a map showing the distribution of concentrations of the elements like aluminum, titanium and chromium (Fig. 6c). Structure examinations by transmission electron microscopy (Fig. 7) show the formation of a new complex phase of a composition similar to $(Al,Cr)_3Ti$ and the local precipitates of Al_2O_3 . The diffraction patterns obtained from the examined area confirm the presence of precipitates within the phase boundary, which suggests that Ti+Cr has “strengthened” the interface.

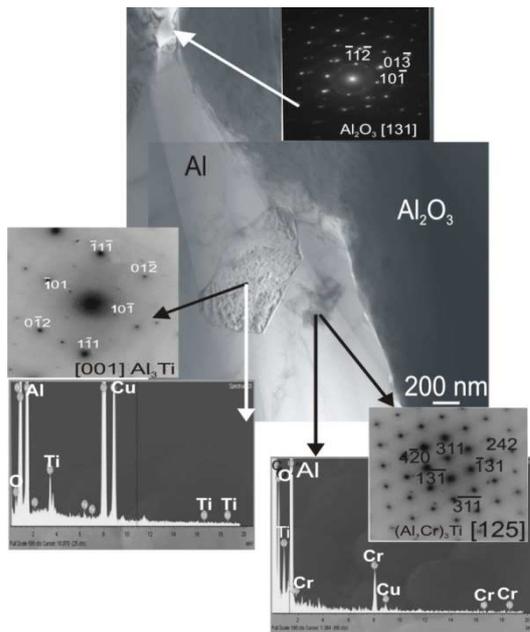


Figure 7. TEM analysis of the $Al_2O_3/Ti+Cr/Al/Ti+Cr/Al_2O_3$ interface of sandwich sample with corresponding EDS spectra taken from different places of the reaction products region and representative selected area diffraction patterns indicate the formation of $(Al, Cr)_3Ti$ phases and Al_2O_3 precipitates.

The results of the investigations described above suggest that the mechanism acting in the examined joints consists in dissolution of the thin layer of Ti+Cr coating into liquid Al which, in turn, results in the formation of new titanium and chromium-containing phases within the metal-ceramic interface. The interfacial chemical reactions contribute in the main part to the adherence of metal/ceramic interfaces. The examined joints have the character of diffusion joints. Chemical and structural changes during wetting and liquid – state bonding in metal-ceramic system indicate, that the significant role of geometrical limit in bonding which can lead to a major difference between chemical composition of interfaces obtained in sessile drop and joining experiment. It shall be highlighted that the geometrical factor impacts the differences in the contact zone structure and mechanical characteristics, since in the wettability test the diameter of a metal droplet is of several millimetres, whereas at liquid-state bonding, Al_2O_3 is bonded with a layer of metal in a form of a foil with a thickness from several dozens to

several hundreds of micrometers. The high surface to volume ratio of the liquid metal makes forming of a layer of reaction products several millimetres thick significantly increase the average concentration of a reactive element in the contact zone (its concentration may vary during joining). Hence it can have a more significant influence on wettability and chemical processes in the contact area.

4. Conclusions

The wetting characteristics of Al on alumina substrates covered with Ti+Cr thin film have been studied by a sessile drop method. Joints of $Al_2O_3/Al/Al_2O_3$ produced by liquid - state bonding of alumina covered with Ti+Cr thin film using an Al interlayer were applied to investigate the mechanism of an effect of the Ti+Cr thin film on the metal-ceramic joining. The following conclusions were drawn:

1. Coating of Ti+Cr can reduce the contact angle between Al and Al_2O_3 to even less than 60° and causes intense chemical reaction proceeding at a relatively high temperature of 1123K.
2. In the process of liquid-state bonding of the alumina with aluminum at $T=973$ K, a 380 nm thick PVD coating of Ti+Cr applied on the ceramic surface at a pressure of 0.01 MPa gave satisfactory results producing a highly reliable bond between Al and Al_2O_3 .
3. Chemical reactions between ceramic and metal during bonding were observed to have a dominant influence on the joints bond strength. The Ti+Cr coating reacts with the metal in the bond layer, causing formation of a complex phase of composition and structure approaching that of $(Al,Cr)_3Ti$, combined with the appearance of Al_2O_3 precipitates, which leads to precipitation hardening of the bonding layer.
4. The Ti+Cr coating has an important influence on behavior in the area of bond interface and in particular on the deformation and failure mode and, as a consequence, on the mechanical stability of joints under operating conditions.

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