Cold Embossing of Ceramic Green Tapes

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Abstract

The development of alumina green tapes, made of water based binder systems, allows the structuring by so-called cold embossing. The cold embossing disclaims time expensive heating and cooling phases as needed for hot embossing. That allows a reduction of process time. The structuring by embossing becomes more practicable for high throughput manufacturing. For investigating the influence of powder amount and with or without plasticizer inside ceramic green tapes on deformation behaviour, tensile tests have been carried out. Furthermore, cold embossing tests with uniaxial compression tools were carried out. Channel structures allow investigations concerning stability against elastic recovery. In a first series of tests various green tape compositions have been investigated. As results inferences to the elastic and plastic material behaviour can be obtained. The characterisation of structured tapes in green state has been carried out by using SEM and scanning surface area by confocal white light microscopy.

Key words: cold embossing, ceramic green tape

1 Introduction

Ceramic substrates are in use for various functional applications, which are produced in multilayer technology. By integrating channel structures or membranes new fields of application can be opened up. Examples include biological and sensor technology or micro reaction systems. Hot embossing of commercially available LTCC tapes is in use as structuring method in the state of development [1 - 3]. The hot embossing process of thermoplastic materials [4] is characterized by a heating period followed by displacement- and forcecontrolled embossing with subsequent cooling phase under constant pressure loading. During heating the polymer becomes flow-able. Finally the embossed part can be removed from the structuring tool. A negative aspect of this technology is the time consuming process time. In this paper the development of so called cold embossing was presented, which is characterized by constant processing at room temperature [5]. Therefore alumina green tapes, made of water based binder systems, have been developed. The use of such binder systems allows an environmental and user-friendly manufacturing of ceramic green tapes. Further the mechanical properties of the tapes and its influence to cold embossing process-ability will be presented.

2 Experimental procedures

2.1 Green tape preparation

For preparing alumina green tapes the powder CT3000SG (Almatis GmbH, Gemany) has been used, a submicron powder with the following particle size distribution values (measurement by

laser diffraction method, Mastersizer, Malvern Instruments, U.K.): $d_{10}=0.18~\mu m,\,d_{50}=0.50~\mu m$ and d_{90} 2.76 $\mu m.$ The measured specific surface (BET-method) is 7.9 $m^2/g.$ The powder was also characterized by SEM studies, as can be seen in Figure 1.

The suspension was prepared by ball milling with alumina grinding balls (10 mm diameter) for 3 hours at 60 rpm. The final slurry was achieved by adding the binder and other organic additives like defoamer as well as surfactant and further homogenisation by ball milling for 16 hours. As binder Duramax B1000 (Rohm and Haas), has been selected in preliminary tests as suitable for subsequent cold embossing. The final slurry was degassed for two hours under pressure of 100 mbar and subsequently for 30 minutes at 50 mbar. Further slips were prepared with addition of glycerine which acts as a plasticizer in the dried green tape.

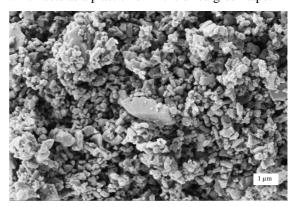


Figure 1: SEM image of the alumina powder CT3000SG, Almatis GmbH.

The rheological characterization has been done by means of a cylindrical measuring system

Z3 DIN. Three alumina slurries with various solid loadings are prepared.

For tape casting experiments a discontinuous laboratory doctor blade tape casting equipment (Netzsch GmbH) was used. The drying was realized under room temperature conditions and low air stream. As carrier foil a polyester film was used. The casting speed was 0.17 m/min. The casting height was set to $800~\mu m$.

2.2 Tape characterization

The tensile testing was carried out with different green tape compositions following EN ISO 527-2. The testing equipment is an Inspekt Retrofit (Hegewald und Peschke GmbH, Germany). Three types of alumina green tapes were analysed by tensile testing with a strain speed of 1 mm/min. For better comparison the tapes have been stored for 16 hours inside a climatic chamber. The used conditions were 50 % humidity and 25 °C. The samples were prepared with special dimension by punching, see Figure 2.

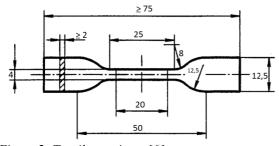


Figure 2: Tensile specimen [6]

2.4 Cold embossing process

The embossing tests have been carried out at a tensile testing machine from type Inspekt retrofit 8562, Instron Deutschland GmbH, Germany with high force allocation. A schematic view of the embossing equipment with inserted guidance system, which was designed and provided by KMS Technology Center GmbH, Dresden, Germany, is illustrated in Figure 3.

The embossing tools have been provided by BPE International Dr. Hornig GmbH, Germany. As testing design a channel structure has been chosen. The manufacturing of this structure was realized by milling. The tool, used for the first series of tests, was made of steel. The channel structure was set to 90 μ m. A technical detail of design is depicted in Figure 4. This structure has been used for screening the ability for embossing of green tapes with different binder and plasticizer content at constant process parameters. The characterization of the tool has been carried out by microscopic investigations like stereo microscopy (Figure 5) and confocal white light microscopy.

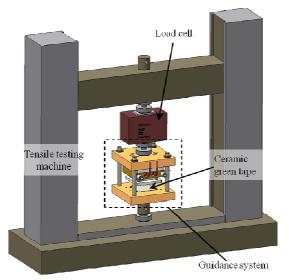


Figure 3: Construction scheme of the embossing equipment with inserted guidance system, designed by KMS Technology Center

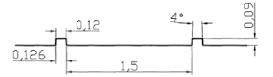


Figure 4: Detail of technical drawing of first embossing tool; made of steel, provided by BPE

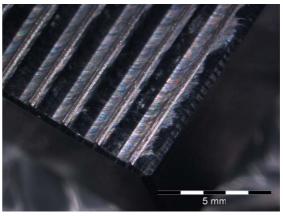


Figure 5: Stereo micrographic image of steel embossing tool, provided by BPE

A second tool was made of brass and the channel structure height was increased to 180 $\mu m.$ A detail of the technical drawing is presented in Figure 6.

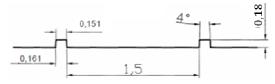


Figure 6: Technical drawing of second embossing tool; made of brass by BPE

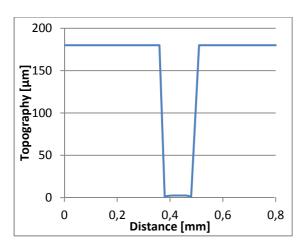


Figure 7: Line scan to replicated channel structure in silicone – here one channel is presented in more detail

The test parameters for the first investigation series were set to 50 N/s speed of indentation with a maximal force loading of 9.5 kN and a dwell time of 5 minutes. The second series of tests were set as follows: speed of indentation was 50 N/s at maximum force loading of 20 kN with a dwell time of 2.5 minutes.

3 Results and discussion

The process-ability of ceramic green tapes during cold embossing has been investigated. The challenge can be seen in the processing at room temperature conditions. This means, no heat treatment was applied for enabling easy gliding of polymeric chains and ceramic particles inside the ceramic green tape because of reaching the glass transition temperature of the used polymer. The first experiments allowed selecting the green tape composition showing the best potential for subsequent structuring processes like embossing. The only difference between these substrates was the use or the absence, resp., of the plasticizer glycerine and its amount in the tape.

Figure 8 shows the results of the tensile testing experiments and its influence on the elastic behaviour. All curves show a linear rising. Here, the green tape was elastically stretched. Afterwards, the material begins to deform, what can be seen in the slower stress implementation. The blue curve shows a green tape made without the use of plasticizer. The maximum stress, in accordance to the following results, is high. At these curves it is noticeable that the maximum value of stress is not the point of break. This is caused by necking of the tensile specimen. The orange curve presents a green tape made with less amount of plasticizer (4.3 vol.-%). That affects a lower maximum stress value in accordance to the tape without plasticizer but a higher elongation can be reached in this way. At last the red curve shows a tape with higher plasticizer content (6 vol.-%). The elongation increases

with higher amount of glycerine while stress decreases.

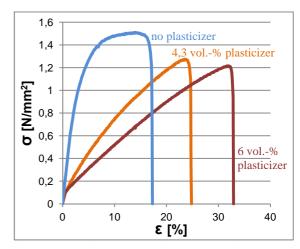


Figure 8: Tensile testing diagram for alumina tapes with constant binder content and with or without plasticizer at green thicknesses of 1.4 mm

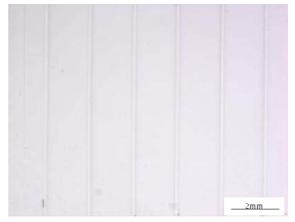


Figure 9: Stereo microscopic image of alumina tape in green state with embossed channel structure

Inside test series No. 1 the embossing tool with structure height 90 μm was used. With this tests the elastic recovery and plastic deformation of tapes has been investigated. The results are illustrated in Figure 10.

In absence of plasticizer the tape shows the highest depth of indentation. The elastic recovery here only was 12 % in length. While material contains a certain amount of plasticizer, the elastic recovery increases in a range of 27 and 52 % respectively. Consequentially, tapes containing a plasticizer will not be used for further embossing tests. The second diagram concerns to the channel depth in relation to the binder content inside the tape. The tape with highest binder content (see red bulk, Figure 11) shows an elastic recovery in range of 16.6 % in length. The difference between tapes with lower binder content around 11 % is not significant.

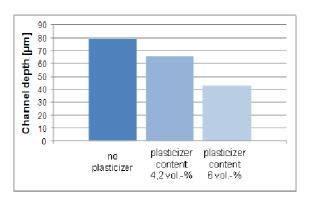


Figure 10: Channel depth inside alumina green tape with or without use of plasticizer, channel height of embossing tool 90 µm

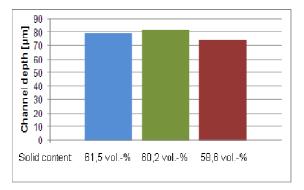


Figure 11: Comparison of embossed channel depth in dependency on the solid content, channel height of embossing tool 90 µm

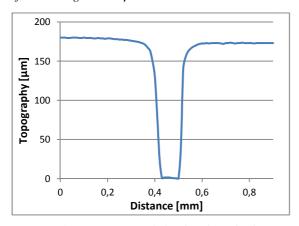


Figure 12: Line scan of final selected alumina composition in green state, embossed with final generation embossing tool, made with confocal white light microscope

As depicted in the line scan of one cold embossed channel (Figure 12 till Figure 14) the structure is not accurately reproduced to the original (Figure 7). Reasons for that can be the elastic recovery of the binder molecules. It can be also noticed that the angle in the changeover between

channel and untreated surface is not clear. Second reason is due to the manually separation of the part. Further, a complete filling of the original surface structure was not reached in the dwell time or the green tape did not adapt accurately to the embossing tool during processing. Additionally pictures can be seen, made with SEM. Here differences of the surface quality are visible.

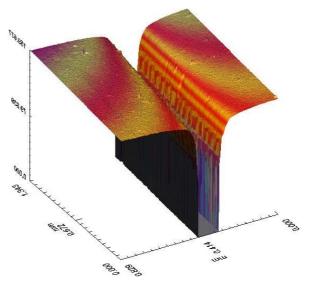


Figure 13: Surface area scan of one channel structure of the embossed alumina green tape, made with confocal white light microscope

While Figure 14 and Figure 15 show a homogeneous surface, in Figure 16 and Figure 17 defects onside the channel structure can be detected. These defects may be caused during tape demolding from the embossing tool. High forces have to be applied to separate the embossed tape, which is caused by seizing tape within micro surface roughness of the tool. This results in the deformation of the whole tape. The highest deformation is located to the center of the embossing tool. This effect is caused by a fixation of the green tape inside the channels itself but also to the smallest roughness of the embossing tool, which was manufactured by milling. By manual separation the direction of force allocation is not reproducible and can destroy the good surface of the embossed part.

Results of such cracked areas are shown in cross section in Figure 16. These cracks are also obvious in the top view (Figure 17). Inside the channel area a large crack can be seen. Furthermore, the bottom chord of the channel is laced with breakouts. Another reason for this behavior can be geometric discontinuity onside the embossing tool, where the green part is indented with.

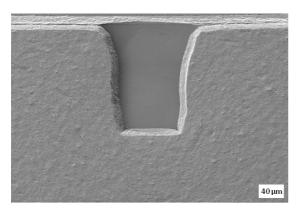


Figure 14: SEM image of a defect free channel structure, alumina green tape without use of plasticizer, solid content 61.5 vol.-%

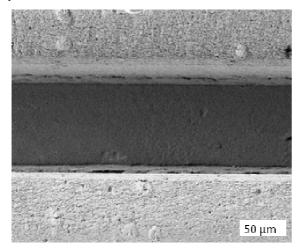


Figure 15: SEM image of structured alumina tape surface in green state, some small defects are visible

4 Conclusion and outlook

Water based binder systems have been used to develop alumina green tapes, which can be processed by embossing at room temperature, socalled cold embossing. The tapes have been characterized by tensile testing. The tapes containing plasticizer show higher elongation than tapes without glycerine as plasticizer. In a first series of embossing tests tapes with and without addition of the plasticizer glycerine and its influence to elastic recovery after embossing have been investigated. Concluding, plasticizer minimizes plastic distortion, because of high amount of elastic behavior. In a second test series tapes with different binder contents and their influence on channel depth were studied. First promising results of channel structures have been presented.

Ongoing tests will allow improving the processability of the final alumina tape composition for subsequent cold embossing. That allows an optimisation of maximum pressure and dwell time under

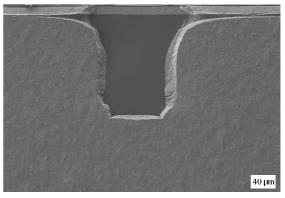


Figure 16: SEM image of channel structure with defects on the side walls, alumina green tape without use of plasticizer, solid content 61.5 vol.-%

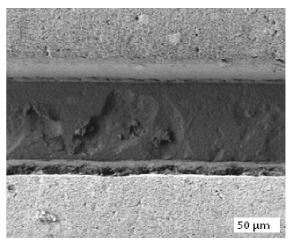


Figure 17: SEM image of structured alumina tape surface in green state with defects onside the channel ground

load. Here the major attention must be taken to the demolding of the parts.

5 Acknowledgements

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6 References

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