Functionally Graded Materials Made by Water-Based Multilayer Technology

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The development of Functionally Graded Materials (FGM) with graded microstructures concerning composition or porosity opens new fields of application. Components with different porosities combine different properties in the gradient structure regarding thermal conductivity and capacity, density, mechanical strength and elastic modulus. Graded microstructures result in innovative, multi-functional properties combinations, such as hard and ductile, electrically or thermally conductive and insulating, magnetic and nonmagnetic for metal-ceramic composites.

The ceramic multilayer technology allows the production of FGM with a high value concerning the degrees of freedom for the designing of FGM. The possibilities of the multilayer technologies were demonstrated by the water-based production of multilayers with graded density (Ca-aluminate/Al_2O_3). Technologies were developed for the production of ceramic platelets as well as the modification of the connection between the different.

SEM-images of cross-sections of the sintered components and of fractured surfaces of tested samples show the different connection between the single layers within the sintered structures as well as the crack propagation.

1 Introduction

In a Functionally Graded Material (FGM) the properties change gradually with position [1] which generates new fields of application. Components with different porosities combine different properties in the gradient structure regarding thermal conductivity and capacity, density, mechanical strength, and elastic modulus which shall result e.g. in improved thermal shock properties [2]. Graded microstructures as combination of two or more materials for example in ceramic-metal composites result in innovative, multi-functional properties combinations, such as hard and ductile, electrically or thermally conductive and insulating, magnetic and nonmagnetic. Possible applications are in a variety of industrial and medical fields, for example as cutting tools, wear resistant components, energy and fuel cell components or as bipolar surgical tools [3–8].

For refractory application the graded porosity can influences the crack resistance in case of thermal shock in a positive way by improving the ratio between initiated stresses and existing strength in each area of the component [2]. A graded porosity in one component leads to lines of tension and increases the resistance to cracks [9]. Furthermore, the pores act as defects which reduce the Young's modulus and can compensate the thermal shrinkage and expansion in the material while application. Compared to dense materials the mechanical strength of porous materials decreases less under thermal shock with respect to initial state. The pore shape and their orientation are important for the mechanical and elastic properties [9, 10].

Several technologies are known for the production of FGM. A very good overview is given by Kieback et al. [1]. Conventional shaping technologies can be used to produce FGM-like powder pressing [11], slip casting [12, 13], powder injection moulding [14, 15], tape casting [16–18] or a combination of conventional shaping technologies like inmould-labeling as a combination of tape casting and injection moulding [19, 20]. However, additive manufacturing technologies can be used for the production of FGM, too, like Laminated Object Manufacturing (LOM) [21] or Thermoplastic 3D-Printing (T3DP) [22–24].

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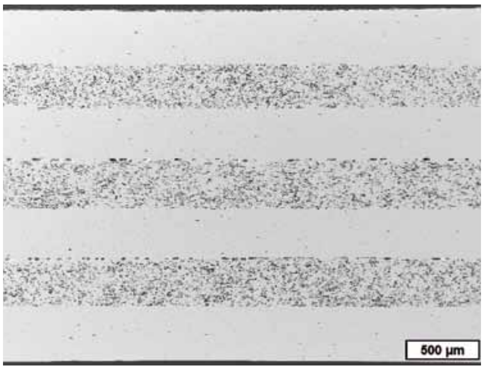

Fig. 1 **SEM-image of cross-section of $MgO–ZrO_2$-multilayer with non-linear graded microstructure (0 %/25 %/…/25 %/0 % porosity) manufactured by multilayer technology**

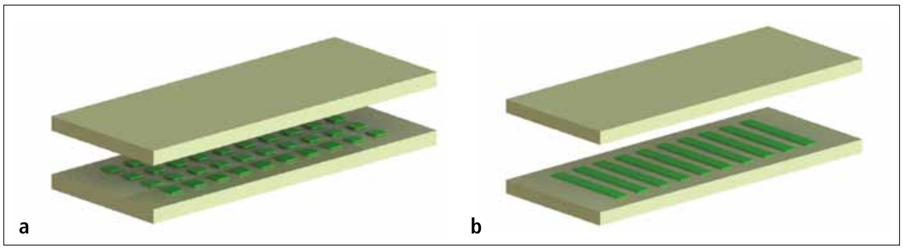

Fig. 2a–b **Selective lamination by dot-shaped deposition of joining suspension (a), and selective lamination by deposition of joining suspension in different lines (b)**

With tape based technologies like multilayer technology or LOM, graded microstructure can be achieved by the combination of single tapes with different compositions [17, 18] (Fig. 1). In a single tape, a variation of the used material, the adjustment of microstructure as well as the adjustment of the porosity by using different particle size distributions or the addition of Pore Forming Agents (PFA) is possible. The connection of the green tapes happens by lamination to create a bulk green component, which is debinded and sintered to one component with graded microstructure and properties. Different lamination technologies are known, e.g. thermocompression [25, 26], cold low pressure lamination [27, 28], or cold chemical lamination [29]. The latter uses organic solvents for the lamination. This process can be optimised if sinteractive-joining suspensions are used to connect the green tapes [16–18].

Ceramic tape casting technology is an established technique for large-scale fabrication of thin substrates [30, 31]. The tape casting process requires a homogeneous, well dispersed suspension which is prepared by mixing and milling the metallic or ceramic powder in a solvent with different organic additives (dispersing agent and binder system), e.g. by using a planetary ball mill. The homogenous, evacuated suspension is cast using a doctor-blade with exactly adjusted casting gap on a carrier foil on a smooth belt. During the subsequent drying the solvent is evaporated and simultaneously the tape thickness decreases. The composition of the tape casting suspension and its rheological properties influence the casting and drying behaviour.

These 2-dimensional structures can be processed by laminating to multilayer structures. This technology is used to produce FGM for applications like porosity-graded piezoelectric ceramics [32], zirconia – stainless steel – composites with graded interface [33, 34], or electrodes for fuel cells [35, 36]. To increase the degree of freedom concerning the composition of one layer the so called side-by-side tape casting technology can be used. This technology allows the variation of the tape composition in one tape by using a doctor blade with different reservoirs for the suspensions, which are cast side by side [37].

But not only planar FGM can be realized with multilayer technology. The so called spiral winding technology, which is known from paper industry and used for spiral wound paper tubes (e.g. for tissue papers) allows the production of cylindrical components by winding and laminating green tapes on a core [38–41]. Several small stripes of green tapes are simultaneously led to the winding spindle and get wound around it [42]. Hereafter, the layers are pressed together by a winding belt. The layers stick together by coating with a joining suspension right before they get to the winding spindle. The main advantages of this process against other winding technologies are the possibility of a continuous production for realizing of a radial graded structure by using tapes with different compositions for each layer.

The aim of the present work was to increase the degree of freedom concerning the composition of FGM during their production by multilayer technology, e.g. to modify the crack propagation. Two different approaches were investigated. First a simple production technology for ceramic platelets was developed. These platelets can be added to the casting suspension and remain into the layer after debinding and sintering. The platelets are oriented because of the shear forces between the doctor blade and the carrier foil and the microstructure of one layer can be graded, e.g. to initiate crack deflection [43–46]. The second approach was to modify the connection between the different layers. This is possible if the used joining suspension is not applied on the entire surface of the tapes but only on selected areas (Fig. 2a–b). Another idea was to laminate pure binder tapes between different green tapes, whereby the pure binder tapes have vias (breakthroughs), which are filled with tape casting suspension (Fig. 3a). After the removing of the binder compon-

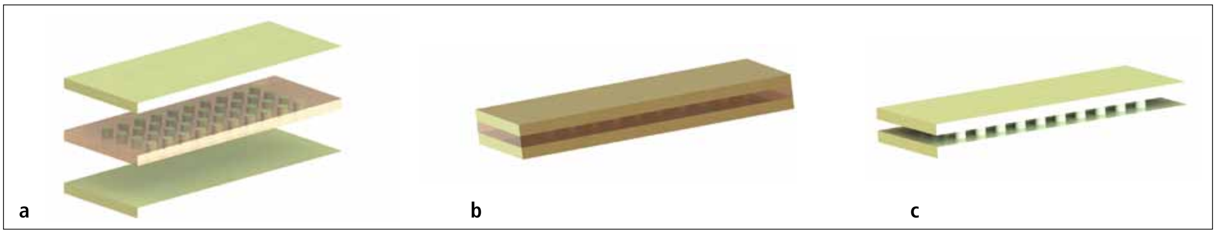

Fig. 3a–c **Pattern of pure binder tape with filled vias between two green tapes (a), pattern of laminated multilayer with pure binder tape in between (b), and pattern of sintered multilayer after removing of organics (c)**

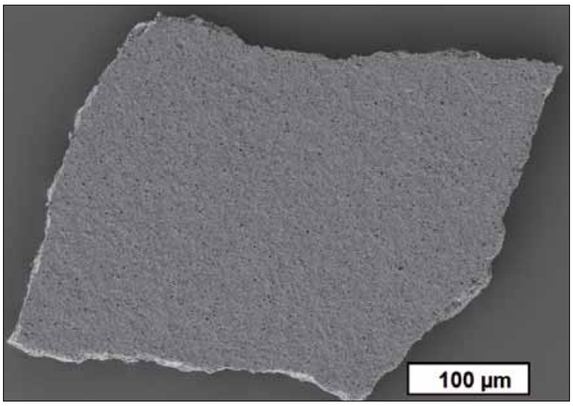

Fig. 4 SEM-image of a single CaAl-platelet

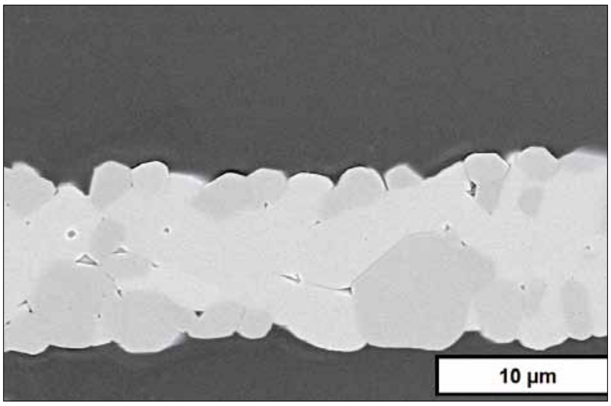

Fig. 5 SEM-image of cross-section of a single CaAl-platelet

ents during the debinding step, big hollow cavities remain and the layers are connected exclusively in the area of the former vias (Fig. 3c).

2 Experimental procedures

2.1 Materials and sample preparation

The used material system was a calcium-aluminate/alumina composite (CaAl), which is formed during the reaction sintering process from starting powders alumina and calcium carbonate. Because of the high porosity, which remain after sintering, the CaAl-system has a good thermal shock behaviour and can be used for e.g. novel kiln furniture [47, 48]. Alumina (CT3000SG, Almatis, d_{50} = 0,4 µm) with a purity higher than 99 % and calcium carbonate (M/Alfa, Scheruhn, d_{50} = 2 µm) with a purity of 99,5 % were used as starting powders for the CaAl-composites. The weight ratio of alumina to calcium carbonate was 9 : 1.

Dispersing agent (Dolapix CE 64, Zschimmer & Schwarz), binder (polyvinyl alcohol: Mowiol 20-98, Kuraray Europe GmbH, and acrylic dispersion: Primal™ ECO 8, Rohm and Haas), plasticizer (glycerine and polyethylene glycol 400), defoamer and surfactant were also added to the suspension [49].

For suspension preparation alumina and calcium carbonate powders were mixed for 1 h in aqueous suspension by planetary ball mill with addition of dispersant and defoamer. The homogeneously and highly dispersed suspension had a solid content of about 80 mass-%. After addition of binder, plasticizer and surfactant the suspension was homogenized in a ball mill for 12 h. After this process the suspension had a solid content of about 60 mass-%. Before casting the suspension was degassed by gently stirring under vacuum (0,1 bar).

For tape casting a laboratory tape casting machine was used which allows the adjustment of the drying temperature and humidity of the airflow over the cast suspension. In this way, skin formation and cracking could be reduced because the drying air aerated with water when it meets the wet suspension film. The suspension was cast on a carrier foil by moving the suspension reservoir and the doctor blade. Between the doctor blade and the carrier foil shearing forces arose, which orientated non-spherical suspension components like pore forming agents or ceramic platelets. Because there was no influence of the casting speed and the casting gap on the orientation of the PFA in the green tape [50], a casting speed of about 4 mm/s and a casting gap of 0,4, 0,6, and 1,2 mm, respectively were used. The thickness of the dried tape varied between 35 – 40 % of the casting gap.

The lamination of the green tapes to multilayers was done at room temperature. Therefor a special sinter-active joining suspension was applied. A roller deposited the suspension as coating on the surface of one green tape. After adding the next tape, a pressure has been applied by using a hand roller or a pair of rolls to connect the lamination partners and to remove the unnecessary joining suspension as well as embedded air.

The thermal treatment steps were carried out under air. For thermal debinding up to 400 °C a heating rate of 0,2 K/min was used. Then the heating rate was increased up to 1 K/min to reach the sintering temperature of 1550 °C. After a dwell time of 3 h passive cooling happened with 3 K/min maximally. The microstructure was characterized by using light and Scanning Electron Microscopy (SEM) to analyse the cross-sections of the multilayers. Non-destructive testing happened by Computer Tomography (CT) and radiography. To characterize the crack propagation the different multilayers were broken in a 4-point-bending-test machinery with outer bearing distance of 40 mm, an inner bearing distance of 20 mm, and a feed speed of 0,05 m/min. To investigate the fracture surface light microscopy, SEM and a topology scanner were used.

3 Results and discussion

3.1 Adding of ceramic platelets

The ceramic platelets were prepared by coating. This technology is known from paper technology and used to modify the surface of paper. Instead of a doctor blade a Mayer rod (wire wound rod) is used, which allow the coating of very thin layers. The same suspension as for tape casting was cast with a thickness of 20 µm upon a carrier foil. After drying the thin layers were debinded and sintered with the same heating and cooling rates like the multilayers, but the sintering temperature was increased up to 1750 °C (200 K more than the normal sintering temperature of CaAl) to get a denser and stiffer structure. After thermal treatment the platelets were disrupted, fractionated (–315 µm), and added to the tape casting suspension.

Fig. 4 shows a SEM-image of a single sintered platelet, which had dimensions of about 250 µm × 200 µm. The sintered platelets had a thickness of about 10 µm

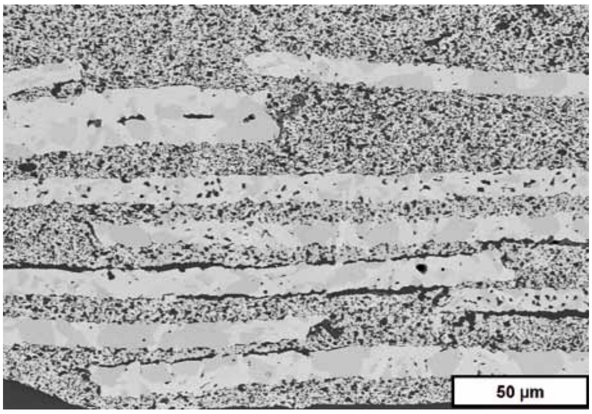

Fig. 6 SEM-image of cross-section of CaAl-multilayer with CaAl-platelets

Fig. 7 Fracture surface of homogeneous CaAl-multilayer

Fig. 8 Fracture surface of CaAl-multilayer with CaAl-platelets

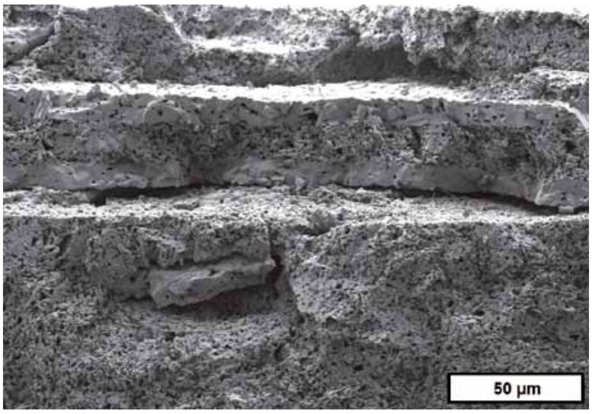

Fig. 9 SEM-image of fracture surface of CaAl-multilayer with CaAl-platelets

and a very dense microstructure (Fig. 5) compared to the CaAl, which was sintered at 1550 °C (Fig. 6). The orientation of the different platelets is almost parallel and corresponds to the casting direction. But also some cracks are visible, which results from the different shrinkage behaviour of the sintered platelets and the non-sintered tape casting suspension during the sintering of the multilayer.

Fig. 7 shows the fracture surface of a sintered CaAl-multilayer which was manufactured by lamination of five single green tapes (without pore forming agents or platelets). Only at the top there is a non-planar area, which probably results from a defect during the lamination of the tapes. If some air bubbles, which are trapped in the joining suspension, could not be removed during the lamination process some pores remain after sintering and the crack is deflected instead of a pure brittle break and a planar fracture surface.

Fig. 8 shows the fracture surface of a multilayer with ceramic platelets. The surface has a much higher roughness which means that the crack was deflected. The platelets show a planar orientation which corresponds to the casting direction. In Fig. 9 a SEM-image of a detail of the fracture surface is shown. The surface looks like stairs and each stair is located at an interface between platelets and CaAl-matrix.

To visualize the rough fracture surface four line scans were made with a contact-free topology scanner. The results are plotted in Fig. 10. The width of the indentations in these curves corresponds to the thickness of the platelets. If a crack runs through the CaAl-matrix and comes to a platelet, the crack is deflected and more energy is needed to arrive the component surface.

3.2 Selected lamination

It is also possible to initiate crack deflection by selective lamination. Different multilayers were produced, but the thickness (400, 600 and 1200 µm, respectively casting gap) and composition (without or with platelets) of the used green tapes as well as the area which was coated with the joining suspension (5 stripes with 2 mm width and 8 mm gap (Fig. 2b), 10 stripes with 1 mm width and 4 mm gap, or fully laminated) was varied. For each type of multilayer 4 – 10 samples were produced and tested by four-point-bending-test. Fig. 11 shows only some of the results. For each type of multilayer one curve was normalized and plotted which represents the behaviour of the whole batch.

The curve for the samples which were fully laminated show a typical behaviour for dense ceramic materials. The force increases nearly linear with the displacement and if a critical stress is reached a catastrophic failure occurs.

The multilayers with the platelets inside show nearly the same behaviour, but the amount of the maximal load is just 60 % of the maximum load of the multilayers without platelets. This results probably from the defects which are created during the sintering at the interfaces between the platelets and the matrix.

All other curves represent samples which were made by selective lamination. The slope of the curves is much lower than the slope of the curve for the fully laminated samples. But after a small decrease of the measured force the force increases again and the catastrophic failure takes place only at significant higher displacements.

3.3 Integration of pure-binder tapes

To check if it is also possible to produce multilayers with a very small connection volume and a large distance between the different layers another type of multilayer was developed. Between two green tapes a pure-binder tape with some filled vias was laminated. The pure-binder tape was cast with the typical suspension composition but without any ceramic powders. After the drying some vias were inserted by punching. Then these vias were filled with casting suspension.

The different tapes were fully laminated by using a sinter-active joining suspension. During the thermal debinding the organic components of the green tapes as well as the organic components of the pure-binder tape were removed.

After sintering a component with planar top and bottom surfaces remained, which were connected only by some small pillar-like structures. Fig. 12 shows an image from the CT and a very good connection between the outer layers and the pillar-like structures as well as the hollow cavities in between. The radiography image in Fig. 13 shows the same.

4 Conclusion

The ceramic multilayer technology enables the production of FGM. Green tapes with different compositions can be prepared, laminated with different technologies, thermally debinded, and sintered to multilayers with inhomogeneous microstructure and with a very good connection between the different layers.

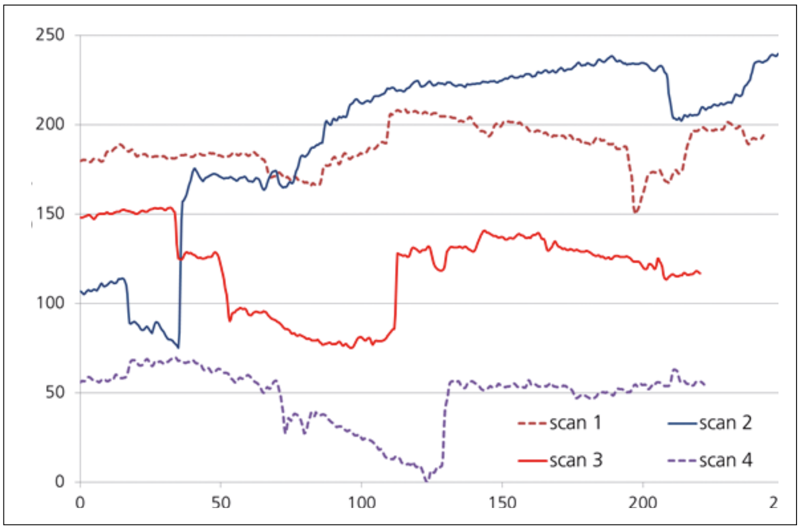

Fig. 10 Results of topology scan of fracture surface of CaAl-multilayer with platelets

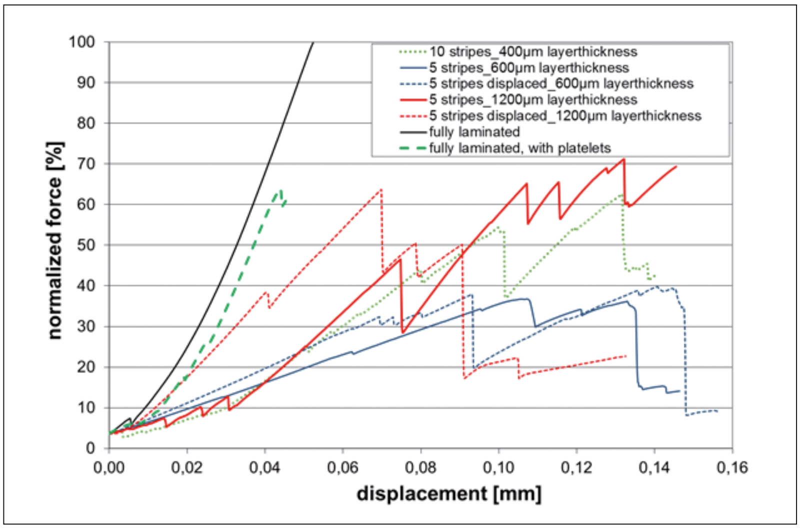

Fig. 11 Generic curves as results of 4-point-bending-tests for different CaAl-multilayers

To modify the behaviour of the multilayers some special structures like platelets can be added to the casting suspension. These platelets are orientated during the casting process because of the shear forces between the doctor blade and the carrier foil. By adapting the coating technology from the paper industry an alternative way for the production of platelets could be established successfully. This process is limited concerning the achievable thickness of the tapes

Fig. 12 CT-image of CaAl-multilayer manufactured with pure binder tape inside

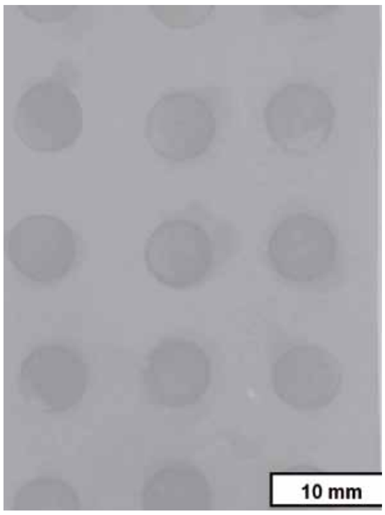

Fig. 13 Radiography-image of CaAl-multilayer manufactured with pure binder tape inside

Tab. 1 Degrees of freedom for the production of FGM by ceramic multilayer technology

Green Tape	Green Multilayer
– Material (kind, amount) – Particle size distribution – Particle shape – Pore forming agents (kind, amount, shape) – Ceramic platelets (kind, amount, thickness)	– Number of tapes – Thickness of tapes – Lamination technology – Lamination areas (fully, selected) – Adding of pure-binder tapes

(>1 µm), but it allows the realization of high aspect ratios (>20) with a high productivity. The specific variation of the connection between the different layers in a multilayer component by the selective lamination enables the modification of the crack propagation behaviour. A brittle fracture can be avoided or shifted to higher displacements.

New degrees of freedom could be accessed for the optimization of FGM. Tab. 1 shows the parameters which can be modified during the production of FGM by ceramic multilayer technology. This multiplicity of parameters requires the development of suitable simulation tools, which can be used to calculate and to optimise the behaviour of the FGM.

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