Fracture Behavior of Plasma Sprayed Ceramic Coatings in Scratch Test†

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Abstract

Fracture phenomena of plasma sprayed ceramic coatings in scratch test were examined. It was found that an inflection point exists in the relation between applied load and friction force. The inflection point shows the critical applied load for flattened particles in a ceramic coating to spall from interfaces. Below the critical load scratch of ceramic coatings is brought by cutting of flattened particles. Over the critical load scratch is significantly brought by spalling of the flattened particles from interfaces.

The critical load for fracture of glass could also be obtained from relation between applied load and friction force. When applied load exceeded the critical load, cracking of glass was clearly observed.

Applying scratch test to sintered ceramics, it revealed that the critical applied load could also be obtained from change of friction force with applied load for sintered ceramics.

The fracture stress of Al_2O_3 coating gives a value of one third sintered Al_2O_3. This result is well consistent with bonding rate of interfaces between flattened ceramic particles and provides further evidence that mechanical properties of a ceramic coating depend on the bonding state at interfaces of flattened ceramic particles.

KEY WORDS : (Plasma Spraying) (Ceramic Coating) (Scratch Test) (Critical Applied Load) (Inflection Point)

1. Introduction

Thermal sprayed ceramic coatings have been continued to increase widely applications in many industrial fields because of excellent wear resistance, corrosion resistance, and thermal resistance of ceramics themselves. However, mechanical properties of a coating are usually much lower than that of sintered material. Therefore, the failure of a coating would occur at a lower loading condition.

When stresses are exerted throughout a coating as in the cases of tensile test, fracture mechanics test[1)] and shear test[2)], it was found that fracture is preferred to occur from interfaces between flattened particles for cohesive failure of a ceramic coating. This fact is well consistent with the finding that the bonding rate between flattened particles is less than one third,[3)] which reveals that the interfaces between flattened particles in a coating are the weakest part in a coating. As a strong inhomogeneous localized stress field is excerted to a coating as in the cases of grinding and cutting, it would be expected that wear of a coating occur through the grooving of flattened particles and their spalling from interfaces.[4,5)] However, it is still not well understood in what way fracture of a coating occurs and how fracture relates to mechanical properties and structure of a coating.

In this report, fracture phenomena occurred in scratching of ceramic coatings were investigated and fracture behavior of the coatings was examined through comparison with these occurred also in scratching of glass and sintered ceramics. A method to evaluate the critical load for fracture of coatings and brittle materials is proposed from the relation between applied load and friction force.

2. Experimental

Ceramic coatings used in this study were Al_2O_3, Al_2O_3-2.3%TiO_2, Al_2O_3-13%TiO_2, Al_2O_3-40%TiO_2 and TiO_2 coatings. These coatings were sprayed onto surface of SS41 mild steel to a thickness of about 250 μm with PLASMADYNE (Mach-1) at a power level of 28kW. The coating surface was then carefully polished.

Glass used was commercially available slide glass. Sintered ceramics were Al_2O_3, Si_3N_4, MgO, ZR-15M (stabilized ZrO_2 with 15mol%MgO) and PSZ(Y_2O_3) (partially stabilized ZrO_2 with Y_2O_3).

Scratch tester (HEIDON-14) used is schematically shown in **Fig. 1.** Load was applied vertically and changed

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by changing weight. Sample attached to sample holder table was driven to move horizontally at a speed of 30mm/min under a certain load. Friction force was recorded with recorder and also digitally memoried at a certain selected distance simultaneously for examining the relation of fluctuation in friction force with fracture in material to be scratched. Scratching was performed with a spherical diamond indenter of a radius of 50 μm.

Scratched surface of sample was examined with optical microscope and scanning electron microscope. The width of scratch was measured by using standard microhardness processor.

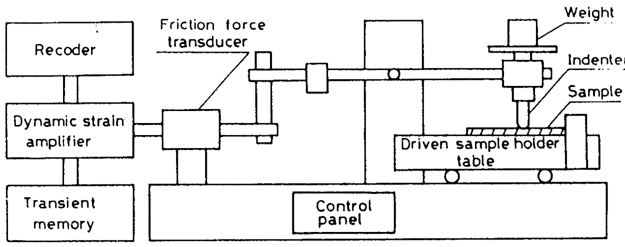

Fig. 1 Schematical diagram of scratch tester.

3. Results and Discussion

Figure 2 shows the typical relation between applied load and friction force for Al_2O_3 and TiO_2 coatings. This reveals clearly that there exists an inflection point in the relation between applied load and tangential friction force. When applied load was lower than that at the inflection point, the friction force increased proportionally to the load. With a further increase in applied load over to the inflection point, the friction force was found to increase more rapidly although friction force increases still proportionally to applied load but at a larger slope. Moreover, fluctuation in the friction force was also significantly different. As shown in **Fig. 3**. little of fluctuation can be recognized below the inflection point, but fluctuation becomes much violent at load range over the inflection point. Examination of scratched coating surface as shown in **Fig. 4** revealed that coating exhibits different fracture behavior. At a load range below the inflection point only grooving of coating particles was found, but when applied load exceeded the inflection point the spalling of particles from interfaces became significant. Therefore, it is evident that the inflection point represents the critical load condition and the load at the inflection point gives critical load for flattened ceramic particles to spalling from interfaces. Valli et al.[6] used the scratch test to evaluate adhesion of thin film and found that changes in friction coefficient indicated critical loads which cause the adhesive fracture of a film. For thermal sprayed ceramic coatings with a lamellar structure, it is reasonable to consider that critical load indicated by the inflection point in the relation between applied load and friction force, which characterizes the cohesive fracture of a coating, corresponds to the result that Valli reported for adhesive fracture of a thin film.

In order to clarify what the inflection point implies, and examine fracture phenomena occurred clearly below and over critical load, glass was scratched under the same conditions as that for coatings. **Figure 5** illustrates the relation between applied load and friction force for glass. The inflection point can also be clearly recognized. **Figure 6** shows typical scratches remained on the glass surfaces

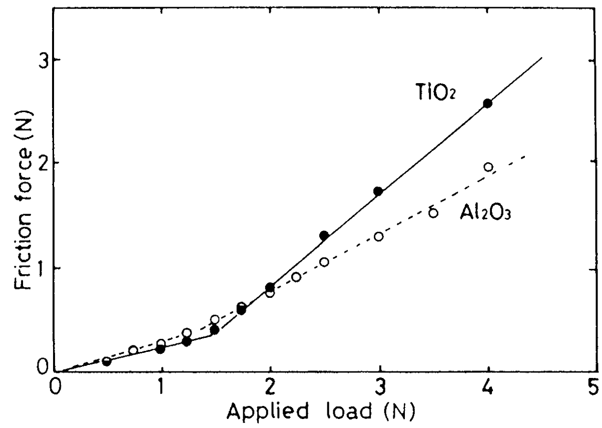

Fig. 2 Relation between applied load and friction force for Al_2O_3 and TiO_2 coatings.

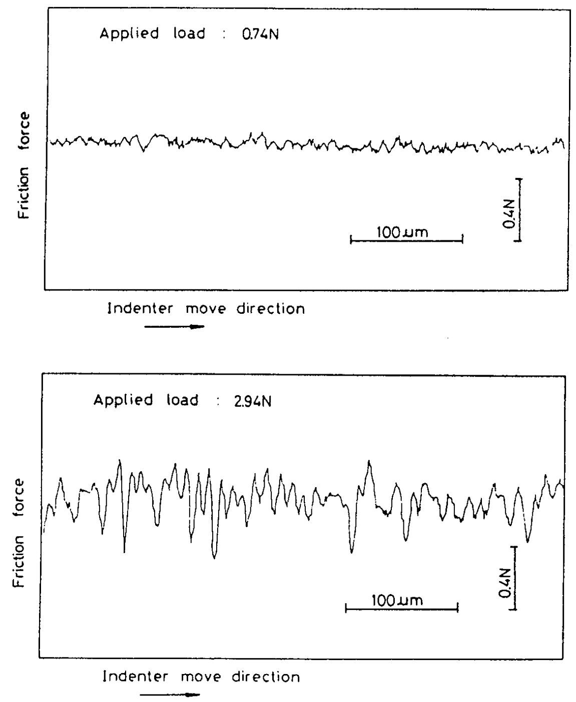

Fig. 3 Comparison of typical fluctuation patterns of friction force at applied load zones lower (top) and higher (bottom) than the inflection point obtained from scratching of TiO_2 coating.

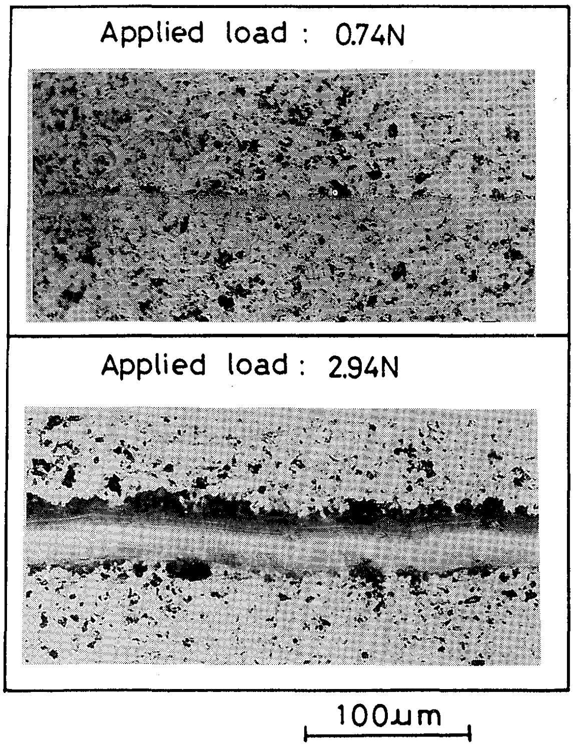

Fig. 4 Typical scratches of TiO_2 coating surface at two load zones lower (top) and higher (bottom) than the inflection point.

corresponding to two different load zones. Below the inflection point, only plastic flow, and no crack were recognized. When applied load exceeded the inflection point, cracking of glass surface was clearly recognized. Examination of scratches by scanning electron microscope showed that the cracks over the inflection point increased with applied load and chipping became significant when the load reached 3.92N. Corresponding to occurrence of cracking, fluctuation in friction force becomes more violent as illustrated in **Fig. 7.** From these results, it is clear that applied load at the inflection point represents the critical load over which fracture occurs during scratching.

The stress distribution when a spherical indenter slides on a flat surface has been analyzed by Hamilton et. al.[7] The maximum tensile stress developed can be obtained as follows.

$$\sigma=\sigma_0\left(\frac{1-2\nu}{2}\right)(1+A\cdot f) \quad (1)$$

where

$$\sigma_0=\frac{F}{\pi a^2} \quad (2)$$

$$A=\frac{3\pi(4+\nu)}{8(1-2\nu)} \quad (3)$$

σ : maximum tensile stress ; σ_0 : average stress over contact area ; F : applied load ; a : contact radius ; f : frictional coefficient ; ν : Poisson's ratio.

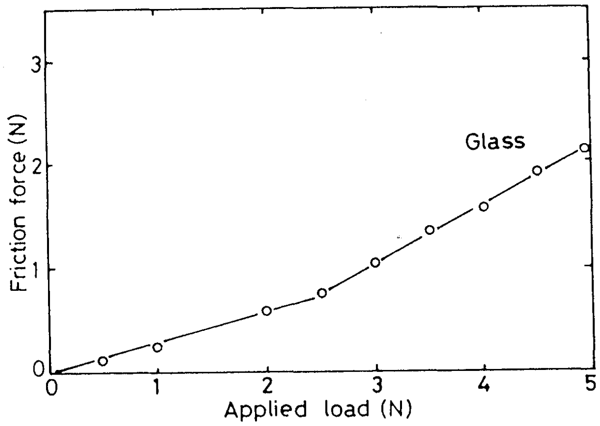

Fig. 5 Relation between applied load and friction force for glass.

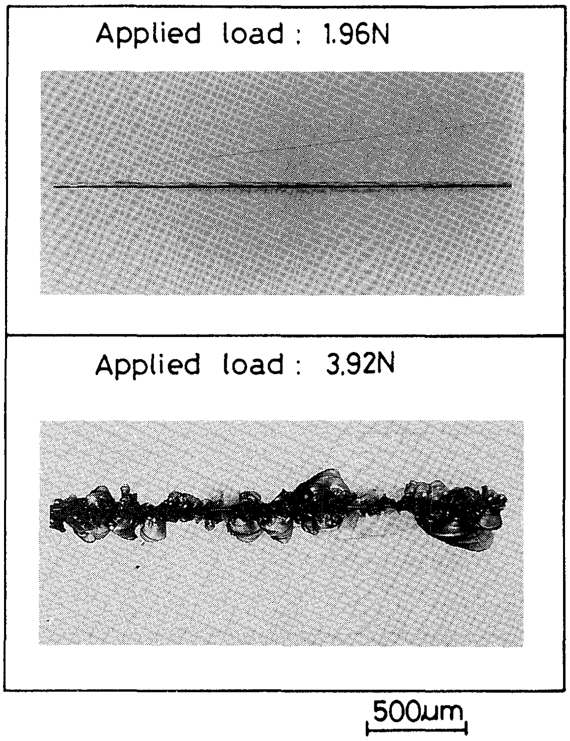

Fig. 6 Typical scratches on glass surface at two load zones lower (top) and higher (bottom) than the inflection point.

Putting half of width of scratch into contact radius, and using critical applied load and frictional coefficient, fracture stress can be estimated from Eq. 1. Using 0.25 as Poisson's ratio of glass, this gives a fracture stress of 2020MPa.

Marsh[8] has studied plastic flow occurred in glass under indentation hardness test by using spherical cavity theory and related the flow stress with hardness by

Young's modulus and Poisson's ratio. From subsequent correlation, he obtained flow stress for various glasses of different hardnesses. Comparison with fracture stress of glass showed that flow stress from the spherical cavity theory represents fracture stress of glass.[9]

Comparing fracture stress of glass obtained above with flow stress (2618MPa or $380 \times 10^3 Lb/in^2$) by Marsh for glass having a same Vickers hardness level (460 H_v) as used in present study (486 H_v), the fracture stress is reasonably consistent with flow stress.[8] These facts suggest that the maximum tensile stress at the critical applied load represents the fracture stress of glass and also flow stress as suggested by Marsh.

Applying the same scratching test to sintered ceramics, similar results were obtained. **Figure 8** shows typical results about relation between applied load and friction force for sintered ceramics. It is clear that the critical load can be obtained from the relation between applied load and friction force for sintered ceramics.

Table 1 and **Table 2** show the critical applied load for ceramic coatings and bulk brittle materials tested respectively. Comparing with bulk materials the critical applied load of ceramic coating is only about half of that of bulk sintered materials. It is considered that this is due to poor bonding state at interfaces between flattened ceramic particles in coating.

The fracture stress obtained from Eq. 1 for ceramic coatings and sintered ceramics tested in present study are also given in Table 1 and Table 2 respectively. In calculation of fracture stress Poisson's ratio was taken 0.25 for sintered ceramics and 0.076 for ceramic coatings.[10]

As suggested by the result for glass and taking the brittle characteristics of all materials used into consideration, it is reasonable to consider that scratching of brittle material can be divided into two zones by critical applied load. When applied load is less than critical load, scratching would be due to plastic grooving. With a further increase in applied load over critical load, cracking becomes significant and fracture occurs. This fact will be much important in processing of brittle materials and ceramic coatings.

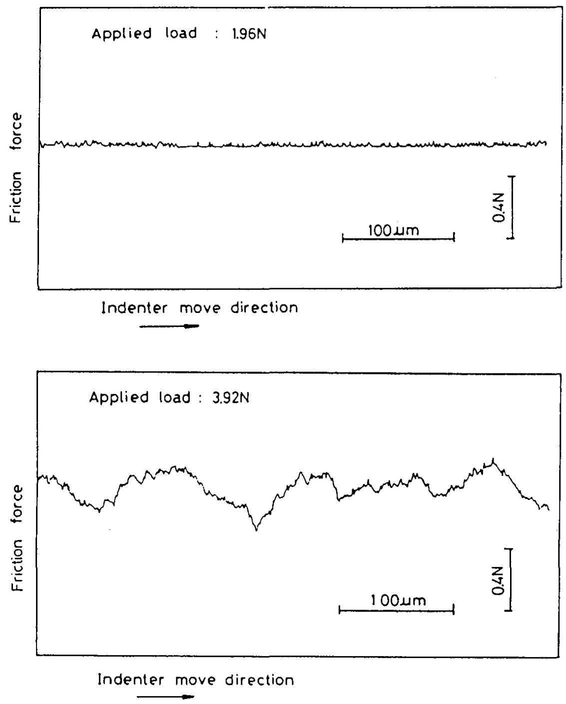

Fig. 7 Comparison of fluctuation patterns of friction force at two different load zones from scratching of glass.

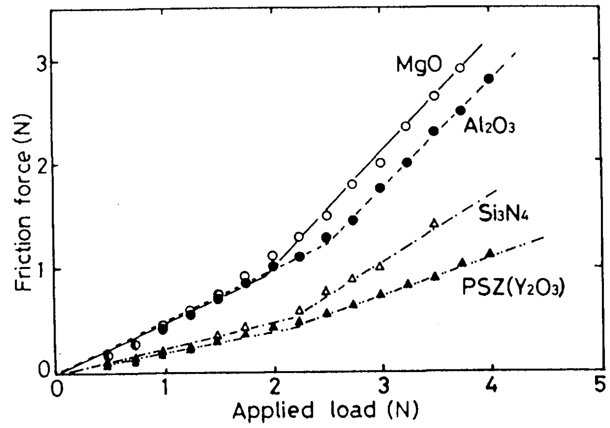

Fig. 8 Relation between applied load and friction force for sintered ceramics.

Table 1 Critical applied load and fracture stress of ceramic coatings

Coatings	Critical load (N)	Fracture stress (MPa)
Al_2O_3	1.22	2618
2.3%TiO_2-Al_2O_3	1.53	2611
13%TiO_2-Al_2O_3	1.35	1814
40%TiO_2-Al_2O_3	0.53	1416
TiO_2	1.31	1883

Table 2 Critical applied load and fracture stress of bulk ceramics

Ceramics	Critical load (N)	Fracture stress (MPa)
Al_2O_3	2.46	7391
Si_3N_4	2.05	6171
MgO	1.88	1661
PSZ(Y_2O_3)*	2.08	4975
ZR-15M**	2.05	3507
Glass	2.35	2023

* : Partially stabilized ZrO_2 with Y_2O_3.
** : Stabilized ZrO_2 with 15mol% MgO.

As shown by **Fig. 9**, fracture stress increases with Vickers hardness. A further examination revealed that fracture stress of ceramic coatings is a little lower than one that would be expected for bulk materials of the same hardness. Therefore, hardness of a ceramic coating would tend to overestimate bonding of flattened particles. This is perhaps due to that hardness of a coating would be dependent on the bonding state at interfaces and hardness of flattened ceramic particles.

For plasma sprayed Al_2O_3 coating with lamellar structure which forms by deposition of flattened ceramic particles, bonding rate between ceramic particles was measured to be less than 32%.[3] This poor bonding limits mechanical properties of the coating.

McPherson showed that the Young's modulus and Poisson's ratio of a Al_2O_3 coating were one fifth and one

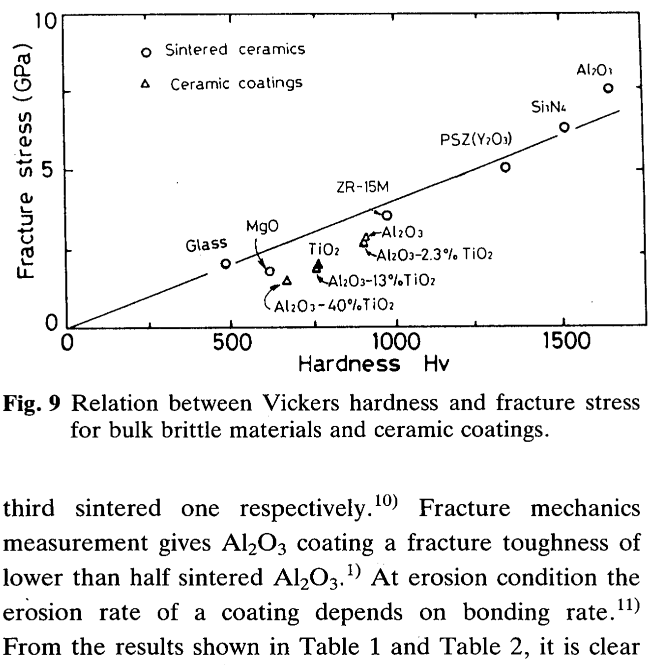

Fig. 9 Relation between Vickers hardness and fracture stress for bulk brittle materials and ceramic coatings.

third sintered one respectively.[10] Fracture mechanics measurement gives Al_2O_3 coating a fracture toughness of lower than half sintered Al_2O_3.[1] At erosion condition the erosion rate of a coating depends on bonding rate.[11] From the results shown in Table 1 and Table 2, it is clear that the fracture stress of Al_2O_3 coating is only about one third of sintered Al_2O_3. All of those results sufficiently show that fracture of a coating will be preferred to occur from interfaces between flattened particles at critical stress when a coating is stressed and mechanical properties of a coating will be dominated by bonding state at the interfaces.

4. Conclusions

Fracture phenomena of plasma sprayed ceramic coatings in scratch test were examined. It was found that inflection point exists in the relation between applied load and friction force. By the inflection point the critical applied load for ceramic particles to spall along the interfaces between flattened ceramic particles can be obtained. Below the critical load scratching of ceramic coatings is brought by grooving of flattened particles. Over the critical load scratch is significantly brought by spalling of the flattened particles from interfaces.

The results of scratching of glass showed that the critical load for fracture of glass could also be assessed from the change of friction force with applied load.

The critical applied load could also be obtained from change of friction force with applied load for sintered ceramics.

The fracture stress of Al_2O_3 coating gives a value of one third sintered Al_2O_3. This result gives further evidence that mechanical properties of a ceramic coating depend on the bonding state at interfaces of flattened ceramic particles.

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