**Cleavage cracking of ductile-metal substrates induced by brittle coating fracture**

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**Abstract**

Brittle coatings are used to protect underlying ductile substrates from damage. Recent experimental observations show that the fracture of a brittle film can cause the micro-cracking of the underlying ductile metal substrate, threatening the continuing safe use of engineering structures. However, the cracking mode of the substrates is unclear and the corresponding mechanism remains poorly understood. Here, by performing room-temperature uniaxial tension experiments with a 10-4 s-1 straining rate, we have observed cleavage cracking in metal substrates (pure iron, AISI 1020 steel and brass) coated with WC-10%Co-4%Cr. Based on the present work, theoretical analysis reveals that the cleavage cracking is the result of brittle coating and its fracture, which synergically inhibit the local plastic deformation of the underlying substrate. One is to restrain the dislocation nucleation and mobility near the interface of the substrate. The other is to bring a local high strain rate loading to the substrate, due to high velocity crack propagation in the brittle coating induced by its fast dynamic fracture. The coupling of these two effects leads to the nucleation of cleavage crack in the normally ductile substrates close to the interface. The onset of cleavage at low strain causes a significant loss in the substrates’ ductility. This detrimental effect will be much more pronounced with the increase in the coating thickness. The present study provides a warning and guide in the design of all brittle coating-metal substrate systems.

***Keywords:***Brittle coating; Ductile metal substrates; Cleavage cracking; Local plastic deformation; Coating thickness.

1. **Introduction**

Generally, coating technology is one of the common surface modification methods to prevent the underlying metal substrates from damage without changing their surface component and structure, therefore improving their service life under harsh environments. Examples include protective coatings to prevent the lucrative engineering materials from wear [1], corrosion [2], high temperature oxidation [3], thermal barrier coatings to improve operating temperature of turbine blades [4], and so on. However, there is often a mismatch between coatings and substrates in terms of microstructures, elastic modulus, and other physical properties. Due to the quite low fracture toughness of brittle films [5], they are easy to fracture when subjected to the external stress [6] or internal stress induced by temperature [3]. Recent experimental observations show that the fracture of a brittle coating can cause the micro-cracking of the underlying metal substrate [7-9]. The presence of these surface microcracks will cause large stress concentrations [10] and introduce stress triaxiality in the substrate [11], which inhibit local plastic deformation of the base metal materials significantly [12], leading to the decrease in materials’ ductility (, elongation to fracture). This detrimental effect will be more pronounced in fatigue cracking [13] and stress corrosion cracking [14] of metals, causing the catastrophic and unpredictable failure of structural components in service. For example, fatigue experiments of harder coating and softer substrate system show that the fatigue crack originating from the brittle coating can propagate through the interface into the substrate, causing a remarkable decrease in substrate’s fatigue life [15-18]. Such significant hazard is the original motivation for the present study to investigate how fracture of a brittle coating causes the cracking of underlying ductile metal substrates.

Previous efforts dealing with the cracking of elastic substrates induced by stiff films are based on a linear elastic fracture mechanics model, claiming that the depth of penetration into the substrate depends on the difference in elastic modulus and toughness between the film and the substrate [19, 20]. However, this model is limited in the cracking of elastic-plastic metal substrates induced by coatings fracture due to absence of the plastic deformation that plays an important role in nucleation of cracks in metals. A new model is required to understand the nucleation of crack in metal substrate induced by a brittle coating fracture. It is well known that the fracture mode of a metal material is usually categorized into brittle and ductile [21], which can be identified from the fractography. A brittle failure is controlled by micro-cleavage crack propagating along a crystallographic plane with high stress concentration at the crack tip. A ductile failure is related to the nucleation and connection of voids, accompanied by a large amount of plastic deformation characterized by dislocation nucleation and dislocation mobility [22]. Therefore, clarifying the fracture mode in the base metal is needed. However, the fracture of brittle films with thickness of several microns could only cause the substrate cracking with a short depth [7-9]. It is difficult to judge the fracture mode from such short cracking lengths, and thus a long fracture depth is needed. It is found that thicker coating has a more pronounced impact on decreasing the fatigue properties of Ti-6Al-4V alloy [15, 16], which indicates that thicker coating fracture may cause longer cracks in the base material.

High velocity oxygen fuel (HVOF) sprayed brittle WC-10%Co-4%Cr coatings are a frequent choice in engineering components, automotive and aerospace industries due to its protection against corrosion and wear. It can remain good quality even at a thickness of several hundred microns [23]. In this paper, the WC-10%Co-4%Cr coatings were sprayed onto iron substrates. The mechanism of brittle coating fracture caused cracking of ductile substrates is proposed based on the fractographies. Associated with that, the dependence of iron substrate ductility on coating thickness is also studied.

1. **Experimental procedures and results**

Three ductile metals with fine ductility were chosen as substrates, i.e. 99.9% pure iron, AISI 1020 steel and brass (64 wt.% Cu and 36 wt.% Zn). We will only introduce the experimental details of iron substrates in the following section, the treatments of the other two substrates are the same. The initial dimensions of the iron substrates were 100 mm × 100 mm × 10 mm. It has been reported that a better interfacial adhesion contributes to the film crack propagates into the substrate, rather than along the interface [24]. Therefore, prior to coatings spraying, grit blasting (GB) pre-treatment was used on the substrate’s surface to strengthen the adhesion by improving surface cleanliness and roughness [25]. However, grit blasting brought in work hardening to the surface and a nano-grained top layer could be obtained with a typical thickness of a few microns [26]. This was also observed in our experiments and confirmed by micrograph in Fig. 1a. The effect of this hardened surface layer on the iron substrates’ ductility first studied by comparison with GB-free iron. The samples were cut into smaller pieces with dimensions as shown in Fig. 1b, and tested under uniaxial tension with a constant strain rate of 10-4 s-1 by using a Letry tensile tester at room temperature. As seen in Fig. 1c, the GB pre-treatment promotes both the yield strength and tensile strength of the iron substrate, but decreases its ductility. The fractographies in Fig. 1d and 1e show that the dimples in the iron substrates transform from well-developed equiaxed-shape to lenticular-shape by GB pre-treatment. The elongated direction of the dimples suggests that the hardened layer with high dislocation density contributes to the formation of microcracks. Thus, this deformed layer should first be removed in order to explore how brittle coating causes the cracking of iron substrate.

The heat treatment was carried out to remove this hardened surface layer [27], to avoid the obvious interdiffusion of elements between the coating and the substrate at the interface, and to prevent crack initiation in the coating. The samples were kept at elevated temperature of 600 °C for 1 h [28]. The annealing processes were performed in quartz tubes with 10−5 Pa pressure to prevent oxidation. As seen in Fig. 2a, the grit blasted iron after annealing (Fe, GB, Annealing) recovers its ductility, showing almost identical behavior with the iron after annealing (Fe, Annealing). Its fractography in Fig. 2b shows that the dimples underlying the deformed surface are equiaxed, indicating that the grit blasting induced hardened layer has been removed. The removal of this deformed layer by annealing can also be observed in coated samples by comparing the corresponding engineering stress-strain curves in Fig. 3a. Of particular interest, the presence of coating about 250 μm in thickness significantly decreases the iron’s ductility. Due to the large thickness of the coating, fluctuations will appear in the engineering stress-strain curve if the coating fractures. As seen in Fig. 3a, the crack density reaches saturated when the applied strain is about 0.1, with a saturated crack spacing of about 0.9 mm. We hypothesize that the coating fracture causes the cracking of iron substrate that decreases its ductility. To validate this hypothesis, the coated iron after annealing is first stretched to , and then unloaded. As shown in Fig. 3b, the depth of the coating crack penetrates into the iron substrate is about tens of microns. These cracks will cause the iron substrate fracture along one of them during tension due to large stress concentration in front of it, making it possible for us to observe the cracking mode from its fractography.

 Fig. 4 displays the fractographies of the specimen in Fig. 3a. It is clearly seen that the cracking mode of the iron close to the interface is cleavage, as shown in Fig. 4a and 4d. As the location away from the interface gradually, the fracture morphology in Fig. 4a-4c transforms from cleavage to quasi-cleavage and eventually to dimples, i.e. from brittle to ductile. The cleavage fracture of the iron substrate is also found in the coated surface, where the upper coating has spalled off during tension, as seen in Fig. 5. In addition, the similar phenomenon is also observed in AISI 1020 steel and brass substrates, as seen in Fig. 6 and Fig. 7. It suggests that the cracking of normally ductile substrates induced by brittle coating fracture is cleavage.

At last, the coating thickness effect on the iron’s ductility loss is also studied. In order to reduce the experimental uncertainties induced by sample preparation, the different thickness of coatings are obtained by polishing the original samples with a thickness about 300 μm after annealing. As shown in Fig. 8, the iron substrates’ ductility steadily decreases as the coating thickness increases.

1. **Discussion**
	1. Cleavage crack initiation in the iron substrate

The fracture of ductile metals is usually accompanied with plastic deformation characterized by dislocation nucleation and dislocation mobility [29]. If the dislocation process at or near the crack tip is inhibited, the fracture manner can be transformed from ductile to brittle [30-32]. This transition is controlled by many factors, such as chemical environment (hydrogen [33] or corrosive media [34]), temperature [31], loading rate [35], stress state [36, 37], and so on. As seen in the Fig. 4a, the cleavage fracture is just limited to the area near the interface. In this section, how the brittle coating and its fracture affect the plastic deformation of the substrates near the interface is discussed.

3.1.1 Short range coating-substrate interaction

As seen in Fig. 3a, 6d and 7d, the presence of coating increases the yield strength of the underlying metal substrates significantly. The involved mechanisms can be summarized into two categories based on the short range film-substrate interaction effect [38-41]. One is to suppress the operation of dislocation sources near the interface, and the other is to prevent the egress of emitted dislocations. Image force due to an abrupt change of shear modulus at the boundary of film and substrate is well known [42]. If a film with a shear modulus *G*f greater than that of the substrate *G*s, would cause a repulsive image force against a dislocation [42-44]. When the surface film is sufficiently thick [45], the image force *f*i per unit length acting on the dislocation in substrate can be expressed as:

 (1)

where *L* is the distance of a dislocation from the film-surface boundary, and *b* is Burgers vector. As revealed in Eq. (1), the image force increases as *L* decreases, which indicates that the effect of image force on dislocations near the interface is remarkable. Fig. 9 shows a schematic illustration of dislocation emission along slip plane when the applied stress increases. If a dislocation is about to nucleate from the dislocation source in the substrate, as shown in Fig. 9a, the repulsive image stress induced by the brittle coating will counteract the shear component of the applied stress, leading to a reduction in the driving force for dislocations emission. The emission of dislocations from dislocation source near the interface can be described by using the concepts developed by Rice and Thomson [46]. The total shear stress, *τ*, on the dislocation is:

 (2)

where *τ*ais the applied shear stress, *τ*i is image stress induced by brittle coating on the dislocation, *τ*f is the frictional stress for the dislocation to move, *τ*p is the image stress of all other dislocations acting on the one, and *N* is the number of emitted dislocations near the dislocation source. If the total shear stress is positive, dislocations will nucleate. As indicated in Eq. (2), the presence of brittle top coating delays the emission of dislocations compared to the free surface.

Both experiments and molecular dynamic simulations have shown that the coating is still able to affect the plastic deformation of substrate, although there is no difference in the shear modulus between them [41, 47-49]. This is attributed to different lattice parameter and crystalline structure of the brittle coating from that of the metal substrate. The brittle coating can serve as an effective barrier for dislocations to escape from the substrate [41, 50, 51]. As a consequence, the emitted dislocations will pile up against the interface along the slip plane to form a one-dimensional array on each slip plane, as schematically illustrated in Fig. 9b. These dislocation pile-ups have two effects on the deformation of the substrate near the interface. One is that producing a back stress (*τ*p in Fig. 9b) to counteract the applied shear stress, which prevents the approach of further dislocations or eventually stop the dislocations generation [52, 53]. The other is that forming high stress concentration at the head of pile-up (*σ*p in Fig. 9b), which contributes to the nucleation of cleavage crack if the normal stress equals to the atomic bonding strength [54].

In addition, because the WC-10%Co-4%Cr coating has a smaller Poisson’s ratio than the iron substrate ([55],[56]), causing tensile stress perpendicular to the loading axis within the substrate near interface under uniaxial tension [57-61]. As schematically illustrated in Fig. 10a, this tensile stress changes the stress state in iron substrate near interface from uniaxial tension (along *z* direction) to biaxial tension (along *z* and *x* direction), thus inhibiting the plastic deformation of substrate near the interface [10]. It will prevent contraction of ductile iron along *x* direction during *z* axis stretching, resulting in the formation of “bending” fractography eventually, as seen in Fig. 10b. As a consequence, the coating’s inhibitory effect on the plastic deformation of iron substrate near the interface can be not only by short range coating-substrate interaction, but also through the difference in Poisson’s ratios between coating and substrate. Under these circumstances, the plastic deformation of the normally ductile substrate will be limited by the presence of a brittle coating, thereby promoting brittle fracture.

3.1.2 Effect of brittle coating fracture on the local deformation of substrate

As shown in Fig. 3b, the location where the iron substrate cracks is exactly below the cracked coating, which indicates the coating fracture induced cracking of the substrate. It is far from being enough to consider only the coating inhibitory effect on substrate in exploring the mechanism of brittle coating induced cracking of ductile substrate. Therefore, the effect of brittle coating fracture on the local deformation of substrate will be discussed in this section. Fast dynamic fracture experiments of brittle materials have addressed that the crack propagated with a sufficient high velocity [62, 63]. In the present study, as a crack nucleates in the brittle WC-10%Co-4%Cr coating under uniaxial tension, it will propagate both towards the interface [64]. This process will inhibit the plastic deformation of the substrate near the interface by introducing a crack with high velocity and locally loading the substrate with high strain rate. It is well known that the crack velocity determines the local plastic dissipation rate in front of the crack tip, i.e. the number of dislocations emitted per unit crack area [65]. In simulations performed by Zhao and Li [66], the number of dislocations emitted from a moving crack is much smaller than that emitted from a stationary crack. This steady state number decreases as crack velocity increases. For a straightly moving crack under mode I loading, the dynamic fracture energy depends only on the crack velocity [67]

  (3)

where *v*c is the crack velocity, *v*R is the Rayleigh wave velocity, and the energy release rate  is the static, time independent energy flux into the crack tip, which depends on the geometry of the specimen and the applied stress *σ*. In Eq. (3), an increasing crack velocity decreases dynamic fracture energy. This indicates that the local plastic deformation of the underlying metal substrate near the interface is suppressed greatly, as the coating crack arrives at the interface.

Of particular interest, the thickness of the brittle WC-10%Co-4%Cr coating is about a quarter of the underlying metal substrates. The instantly fast fracture of the coating will result in the local straining of the substrate with an extremely high rate during a short time (equals to the time that the crack propagates in the coating needed). A large amount of experimental work have shown that increasing strain rate has the same effect as lowering temperature in inhibiting the plastic deformation of materials, i.e. promoting their ductile-to-brittle (DBT) transition [68-74]. According to the rate-controlling deformation mechanism [74, 75], the strain rate effects on the flow stress under uniaxial tension is given by

 (4)

where  is a pre-factor, is the Gibbs free energy of activation for the stress-assisted, thermally activated process, *k* is the Boltzmann constant, and *T* is the temperature.  and *V* are the activation energy for overcoming the obstacles to dislocation motion and the effective stress activation volume, respectively.  is the thermal component of the total stress, i.e. accounts for the stress needed to overcome the short-range barrier responsible for the temperature and strain-rate dependence. The applied shear stress *τ* has two components:

  (5)

is the athermal contribution to the flow stress, resulting from long-range internal stresses impeding the plastic flow, and its temperature and strain-rate dependence is weak. The shear stress *τ* and its components are related to the normal stress *σ* and its corresponding components by the Taylor orientation factor [74]. As seen in Fig. 3a, new cracks continue to appear in the coating until the applied strain reaches about . At smaller applied strain (initial stage of work-hardening), the rate-controlling deformation mechanism is attributed to the emission of dislocations from new slip planes in the single slip system [76]. Molecular dynamic simulations on the emission of dislocations in front of mode I crack, shows that as the straining rate rises, the critical stress intensity for dislocation nucleation increases [77]. At larger strain (later stage of work-hardening), the mechanism is then attributed to the intersection of dislocations [75, 76]. The rapid increase in dislocation density at high strain rate increases the dislocations motion resistance, resulting in the fact that the plastic deformation cannot fully proceed, thereby limits the defromation.

In order to clarify the contribution of the coating inhibitory effect and its fracture effect to the substrate ductility, the coating was first pre-notched. For ductile substrate supported brittle coating under uniaxial tension, it reported that the tensile normal stress is maximum at the mid-point of the still adhered segment of the coating [58, 78]. When the stress exceeds the fracture stress, cracks will initiate at the middle of the attached coating segment, leading to the same crack spacing, until the crack density becomes saturated. Therefore, as seen in Fig. 11a, the coating was pre-notched with about 0.7 mm interval (less than the saturated crack spacing), to make sure no cracks will appear in the coating during tension. The engineering stress-strain curve of sample after pre-notch shown in Fig. 11b indicates that the coating inhibitory effect (strengthening effect) on the substrate still remains, and it decreases the ductility of the substrate. The contribution of coating fracture effect to the substrate’s ductility can be identified by comparing the engineering stress-strain curves of “Fe, GB, Coating, Annealing, Pre-notch” with “Fe, GB, Coating, Annealing”.

As described by Kelly et al, the ratio of the maximum shear stress to the maximum tensile stress () determines whether the failure of a ductile material in front of crack tip is shear or cleavage [79]. The shear stress controls emission and slip of dislocations, thus blunting the crack. However, the normal stress contributes to the initiation and growth of cleavage crack [79]. As shown schematically in Fig. 9, the normal stress on the substrate near the interface will increase remarkably, due to dislocation pile-up induced by brittle coating. On the other hand, the dislocation pile-up and repulsive image stress induced by the brittle coating will also counteract the shear stress on dislocation sources. Combining the constraint effects on plasticity induced by local high strain rate loading, high velocity crack propagation and the difference in Poisson’s ratios between coating and substrate, the cleavage cracking is preferable in the normally ductile metals close to the interface, as shown in Fig. 4, 6 and 7.

* 1. Coating thickness-dependent ductility of substrates

The above discussions in section 3.1 have expounded the mechanism of how a brittle coating causes the nucleation of cleavage crack in the iron substrate. In this section, we will discuss the coating thickness dependence on irons’ ductility, i.e. how long the initial cleavage crack can propagate in substrates under different coating thickness. Considering the initial cleavage crack propagates dynamically in the iron substrates under tension, the driving force is its kinetic energy and the work done during the crack extension by the applied stress. At the instant the crack starts to move in a cleavage mode, the time required for the external load to sense the crack motion is given by,  ( is the half-length of the sample) [49]. The total crack growing time, , is given by  where  is the crack velocity and is crack propagation distance (less than 100 μm in the present study). Assuming that the microcleavage crack moves at speeds of at least , then  is about two orders of magnitude smaller than , meaning that the applied stress doesn’t contribute to the cleavage crack growth. Therefore, the energy required to drive the cleavage crack propagation only comes from its kinetic energy. The kinetic energy of a dynamic brittle crack per unit width is given by [80]

 (7)

Where  is the critical size of the cleavage crack,  and  are the Young's modulus and density of the substrate, respectively,  is the initial velocity of the cleavage crack, is a dimensionless constant, determined from , and  is the stress for cleavage crack nucleation. Here, we assume that the critical sizes of the cleavage cracks induced by the coating fracture in iron substrates are the same, but with different initial velocities under different coating thickness. The plastic energy induced by emitting dislocations and the surface energy induced by forming new surfaces provide resistance to crack propagation [49]. As discussed in section 3.1.2, the crack velocity determines the plastic energy dissipation by affecting the number of dislocations that can emit. In addition, if the crack moves fast enough, the motion of dislocations may be slower than the crack and some of them are annihilated at the crack surface [51]. As a consequence, these emitted dislocations cannot reduce the kinetic energy of the moving crack additionally via elastic interactions (known as “shielding effect” [8]). The higher the initial crack velocity, the larger the driving force and the smaller the resistance to the cleavage crack propagation, and thus the longer the distance that it can propagate in the substrate. Therefore, the initial velocity (or kinetic energy) of the cleavage crack determines how far it will extend under its own momentum.

 As discussed in 3.1, the fracture of brittle WC-10%Co-4%Cr coating causes the nucleation of cleavage crack in iron substrate, that is to say, the kinetic energy of the moving coating crack accounts for the increase of energy in substrate. Assuming that there is no energy dissipation along the interface when the coating crack arrives at the interface, according to the energy conservation principle, it can be given by

 (8)

where the energies refer to the energy per unit width.  is plastic energy dissipation in the substrate before the cleavage crack nucleation, which depends on the coating crack velocity.  is the surface energy dissipation when the cleavage crack nucleates in the substrate (,  is surface energy of the substrate per unit area).  is kinetic energy of the coating crack as it approaches to the interface, which can be expressed as:

  (9)

where Z is a dimensionless parameter, depending on the elastic mismatch between coating and substrate [64]. , , , , and  are the Poisson’s ratio, fracture stress, Young's modulus, surface energy per unit area and thickness of the coating, respectively. Inserting the kinetic energy of coating (the expression is the same as substrate’s, as described in Eq. (7) ) to Eq. (9), the crack velocity as it leaves the coating is

 (10)

As indicated in Eq. (10), the higher the coating thickness, the larger the crack velocity as it approaches to the interface and the larger the local loading rate to the substrate, hence, the smaller the plastic energy dissipation in the substrate before the cleavage crack nucleation and the larger the kinetic energy of the initial cleavage crack. Therefore, the ductility of the iron substrates decreases with the increase in the WC-10%Co-4%Cr coating thickness, as seen in Fig. 8.

1. **Conclusions**

The phenomenon that brittle film induced cracking of ductile substrate causes enormous threat to the continuing safe use of coated ductile materials. To clarify the mechanism of this phenomenon, good coating adhesion is necessary. In the present study, grit blasting pre-treatment is used to improve the WC-10%Co-4%Cr coating adhesion to substrates. However, the formation of work hardened surface layer induced by this pre-treatment contributes the nucleation of microcracks, resulting in the decrease in substrates’ ductility. This deformed surface layer is first removed by annealing at 600 °C for 1h. Cleavage fracture of the ductile substrates (pure iron, AISI 1020 steel and brass) induced by WC-10%Co-4%Cr brittle coatings cracking is observed under uniaxial tension experiments with a 10-4 s-1 straining rate at room-temperature. Based on the present work, a new model has been proposed. It reveals that the cleavage cracking is the result of brittle coating and its fracture, which synergically inhibit the local plastic deformation of the underlying substrate. One is to restrain the dislocation nucleation and mobility near the interface of the substrate through short range interaction and difference in Poisson’s ratios between coating and substrate. The other is to bring a local high strain rate loading to the substrate, due to high velocity crack propagation in the brittle coating induced by its fast dynamic fracture. The coupling of these two effects leads to the nucleation of cleavage crack in the normally ductile substrate close to the interface. The onset of cleavage near the interface at low strain causes large stress concentrations in the substrates, resulting in a significant loss in ductility. The decrease in the substrates’ ductility dependence on the coating thickness is also studied. According to energy conservation principle, thicker coating fracture initiates cleavage crack in the substrate with a larger the kinetic energy, resulting in a more pronounced decrease in the ductility. The present study has clearly stated the mechanism of brittle coating induced cracking of ductile substrate, which will provide a warning and guide in the design of all brittle coating-metal substrate systems.

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