

# Iridium Coating: Processes, Properties and Application. Part I

## Processes for protection in high-temperature environments against oxidation and corrosion

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The successful use in rocket engines of iridium as a barrier coating is an important area of high-temperature application. The Ir coating must be continuous and dense in order to protect the underlying material from corrosion and oxidation. The microstructure and morphology of the coating can be effectively controlled by varying the deposition conditions. The microstructure has an important influence on the physical and mechanical properties of the coating. A number of deposition processes, which have different conditions and requirements, have been employed to produce Ir coatings on various substrate materials.

Part I of this paper presents the introduction and reviews the different deposition processes, while Part II will deal with texture and structure evolution, mechanical properties, growth mechanisms and applications of Ir coatings. The mechanisms of micropore formation after high-temperature treatment will also be investigated in some detail.

### **1. Introduction**

Platinum group metals (pgm) have attracted increasing attention due to their high melting points, excellent catalytic activity and superior resistance to corrosion (1). Both Pt and Ir can be used as protective coatings in extreme environments due to their high resistance to corrosion (2). Ir exhibits the highest resistance to corrosion in molten oxides among metals. Owing to its high cost, low catalytic activity above 800°C and low hardness, the use of pure Pt in high temperature applications is limited. Small amounts of Ir and ruthenium are commonly added to Pt to obtain a harder and stronger alloy that retains the advantages of pure Pt. Due to the high cost of the bulk metals, Pt and Ir are often replaced with thin metal layers deposited onto cheaper supports. Depositions of noble metal thin films are of particular interest because of their unique physical and chemical properties. Ir is a promising candidate for a wide range of applications due to its

high melting point, low oxygen permeability, high chemical stability and superior oxidation resistance.

### 1.1 Properties and Behaviour of Iridium

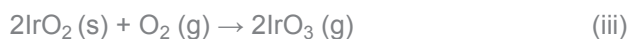
Ir is the sole metal that can be used as a container material at  $\geq 1800^\circ\text{C}$ , although its resistance to oxidation in oxygen environments is poor at these temperatures. Therefore, Ir crucibles are only exploited in inert environments (3, 4). Meanwhile, an Ir film can act as an effective barrier to carbon diffusion (5–7). Carbon contamination of Ir is undesirable as it makes it practically undeformable due to brittle intercrystalline fracture (8). The deformation behaviour of Ir is sensitive to oxygen at elevated temperatures, however Ir is the only metal to have superior high-temperature mechanical properties including creep properties in an inert environment (9, 10). Ir displays poor workability even at elevated temperatures and this substantially limits its industrial applications.

Ir has a face-centred cubic (fcc) structure and behaves like a fcc metal under high temperatures in comparison with such refractory body-centred cubic (bcc) metals as tungsten, niobium and tantalum (11). Bulk Ir shows a ductile-to-brittle transition and exhibits cleavage fracture under tensile conditions (5, 12, 13), although the type of cleavage depends on the impurity content. Carbon- and oxygen-free metal always shows transgranular cleavage despite considerable plasticity, while contaminated Ir is undeformable and its fracture mode is intergranular cleavage or brittle intercrystalline fracture (14). Monocrystalline Ir exhibits high plasticity, but cleaves under tension at room temperature, while polycrystalline Ir displays brittle fracture over a wide range of temperatures (15–21). The brittle nature of Ir remains a puzzle. Some physical properties and their general agreement with empirical cleavage criteria permit a qualitative indication that brittle fracture is an intrinsic property of fcc Ir. These physical properties are elastic moduli, which are unique for a fcc metal (22). Their formal substitution into empirical cleavage criteria including the Rice-Thomson (R-T) criterion leads to the conclusion that Ir is an intrinsically brittle fcc metal despite its high plasticity (23). However, no detailed mechanism has been proposed.

The plasticity of carbon- and oxygen-contaminated polycrystalline Ir is close to zero and does not depend on grain size. The brittle intergranular fracture encountered in polycrystalline Ir was believed to be caused by impurities (16–30). However, Panfilov (20), and Hecker, Rohr and Stein (31) suggest that it is intrinsic to high purity Ir. Lynch

(32) discussed possible reasons for the anomalous cleavage fracture in fcc Ir and suggested that cleavage occurs by an alternate-slip or nanovoid coalescence process. The anomalous fracture behaviour is probably associated with unusual crack-tip surface structure and bonding characteristics rather than with some unusual bulk property. The occurrence of brittle fracture in Ir is thought to be related to the energetics of the dislocation core, in particular the extremely high unstable stacking energy (33–35). Cawkwell *et al.* (36) studied the origin of brittle cleavage in Ir by atomistic simulation using a quantum mechanically derived bond order potential and suggested two core structures for the screw dislocation, a glissile planar core and a metastable non-planar core. Transformation between the two core structures was athermal and led to exceptionally high rates of cross slip during plastic deformation. Associated with this athermal cross slip was an exponential increase in the dislocation density and strong work hardening from which brittle cleavage was a natural consequence. A polycrystalline Ir coating prepared by double glow plasma (DGP) fails predominantly by grain boundary brittle fracture at room temperature. This intergranular fracture in polycrystalline Ir coating may arise from low cohesive strength of the grain boundaries (12). Doping with thorium, cerium and W increases the ductility of Ir and its alloys and suppresses grain boundary fracture, which can be used in some high-temperature structural applications (37–42).

According to the empirical rule for a metal,  $T_{\text{re}} = \frac{1}{2} T_{\text{mel}}$  (where  $T_{\text{re}}$  is the recrystallisation temperature and  $T_{\text{mel}}$  is the melting point),  $T_{\text{re}}$  for Ir should be about  $1200^\circ\text{C}$ .  $950^\circ\text{C}$  is the lowest temperature for recrystallisation reported from experiments with highly pure polycrystalline Ir (43). At low temperatures in air, Ir oxide ( $\text{IrO}_2$ ) film is formed, but at temperatures above  $1100^\circ\text{C}$ , the Ir oxides decompose and the surface remains bare (44). Volatile  $\text{IrO}_3$  is formed at  $1100^\circ\text{C}$ . The oxidation rate of Ir at  $1800^\circ\text{C}$  in still air at one standard atmospheric pressure is  $\sim 1 \mu\text{m h}^{-1}$ ; when the temperature is increased to  $2200^\circ\text{C}$  the oxidation rate is increased ten times (45). The reactions of Ir with oxygen are thought to proceed as in Equations (i) to (vi) (6, 46, 47):





**Table I** summarises the properties of Ir.

## 1.2 Applications of Iridium Coatings

The pgms are potential diffusion barriers. Pt, Ru, Ir, rhodium, osmium and rhenium have been investigated as potential diffusion barriers for carbon (55). Despite the disparity in solubility, there is significant evidence that carbon diffuses rapidly in Pt precluding its application as a discrete diffusion barrier. Re does not form carbides and has low carbon solubility. Rh and Ru display slightly lower carbon solubility, with Ir having the lowest carbon solubility. Os can perform as an environmental barrier similarly to Re and Ir. Pt, Ir and Os have served as oxidation resistant surface coatings, which can be applied as interdiffusion barriers for coatings and composites in high-temperature material systems.

Owing to its unique physical and chemical properties, Ir has been evaluated for applications in a wide range of fields, including as a barrier layer on structural

carbon materials (56, 57), Ir crucibles (58), Re-Ir rocket thrusters (59), heavy metal ion sensors (60), precision glass moulding components (61, 62), patterned thin film microelectrodes (63), spark plug electrodes (64), microelectronics (65) and optical industries (66, 67). Some other applications include as a barrier material in microelectronic devices, protective coatings for some electrode materials and moulds (62), as well as the removal of carbon monoxide contaminants from hydrogen in automotive pollution control (68). Ir and its alloys can be used as anti-stick coatings for glass moulding processes (62, 69–73). An amorphous alloy of Re-Ir-Nb prepared by a sputtering method has been used as a release film for a moulding die (74). Epitaxially grown Ir films on  $\alpha$ -alumina were successfully utilised as intermediate electrodes for epitaxial growth of aluminium nitride (AlN) films for electronic device applications (75). Ir coatings have been used as electrical contacts in oxide high-temperature superconductors and as anti-corrosive coatings for anodes in the electrolysis of seawater (76, 77).

Due to its high catalytic activity, Ir is also used as a component of binary and ternary alloy anodes for the direct oxidation of methanol, ammonia and acetic acid synthesis. Furthermore, Ir can be used in rocket combustion chambers, fuel containers for nuclear power in space, radiation sources for medical treatment and engine ignition devices. One of its more exotic uses

**Table I Properties of Iridium**

Property	Value	Ref.
Melting point	2447°C	(5)
Oxygen permeability	$<10^{-14} \text{ g cm}^{-1}\text{s}^{-1}$ (2200°C)	(48)
Oxidation rate <sup>a</sup>	$\sim 1 \mu\text{m h}^{-1}$ (1800°C)	(45)
	$\sim 10 \mu\text{m h}^{-1}$ (2200°C)	(45)
Density	22.562 kg m <sup>-3</sup>	(49)
Vickers hardness	3.2 GPa	(50)
Thermal expansion coefficient	$\sim 6.2 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$	(51)
Thermal conductivity (0–100°C)	1.48 J cm <sup>-1</sup> s <sup>-1</sup> °C <sup>-1</sup>	(52)
Specific heat (0–100°C)	0.134 J g <sup>-1</sup> °C <sup>-1</sup>	(52)
Electrical resistivity	5.1 $\mu\Omega$ cm	(53)
Young's modulus	520 GPa	(54)
Tensile strength (annealed), 20°C	490–740 MPa	(52)
Poisson ratio	0.26	(52)

<sup>a</sup>Coating, in still air at 1 atm

is as a container for the plutonium oxide fuel cladding material in radioisotope thermoelectric generators – the major source of onboard electric power in spacecraft sent to explore the outer planets (21). The Ir-Re rocket chamber is also a successful application, allowing an increase in satellite life from 12 to 15 years and gaining US\$30–60 million in added revenue per satellite (78).

### 1.3 Failure Modes and Protection of Iridium Coatings

Ir coatings tend to fail at high temperatures through an oxide sublimation mechanism. Above 1100°C, formation of gaseous IrO<sub>3</sub> results in active surface oxidation in air. **Figure 1** shows the oxidation behaviour of a DGP Ir coating debonded from a graphite substrate heated in air. An endothermic reaction was observed at 1074°C as shown in **Figure 1(a)**, consistent with the formation of IrO<sub>3</sub> at 1100°C. The weight changes are shown in the thermogravimetry (TG) curve (**Figure 1(b)**). A mass gain was observed above 800°C followed by a significant mass loss above 1227°C. The mass gain and loss were due to the formation of solid IrO<sub>2</sub> and gaseous IrO<sub>3</sub>, respectively. The mass changes corresponded to Ir oxides formation.

Typically, alloying additions (73, 79) or oxide overcoats (80, 81) are used to mitigate sublimation losses. Sublimation losses may not be a real concern in the inert gas coolant chosen for a space reactor. Composite coatings may however endure higher service temperature and could ensure longer service life for the

Ir coating, providing better protection for the substrate from oxidation at high temperature. Refractory oxide coatings such as zirconium dioxide (ZrO<sub>2</sub>), hafnium(IV) oxide (HfO<sub>2</sub>) and Al<sub>2</sub>O<sub>3</sub> have been produced on an Ir coating surface to inhibit the evaporation and oxidation of Ir and seal the micropores in the coating (78, 82). An Al<sub>2</sub>O<sub>3</sub> layer was formed on the surface of a magnetron sputtered Ir-Al composite coating after oxidation, which could improve the oxidation resistance of the coating (83). Ir-Al forms an Al<sub>2</sub>O<sub>3</sub> layer, effectively suppressing excessive surface oxidation. An Ir aluminide alloy or Ir with a surface aluminide seems favourable as an oxidation and diffusion resistant coating. At present, Ir-based alloys, Ir-M (M = Ti, Nb, Hf, Zr, Ta and V), have gained attention as new high-temperature materials due to their high melting points and superior oxidation resistances (84). Ir-based alloy coatings such as Ir-Al (82), Ir-Ta (85), Ir-Pt (86), Ir-Ni (87) and Re-Ir-based alloy coatings (74) have also been investigated as high-temperature oxidation coatings. There is much interest in Ir as a barrier coating in advanced materials for high technology applications.

## 2. Processes of Iridium Coating

For the coating to adhere well to the substrate, the coating should have a similar coefficient of thermal expansion to that of the substrate, or form a strong chemical or metallurgical bond with the substrate at the interface (88). A dense layer is required for

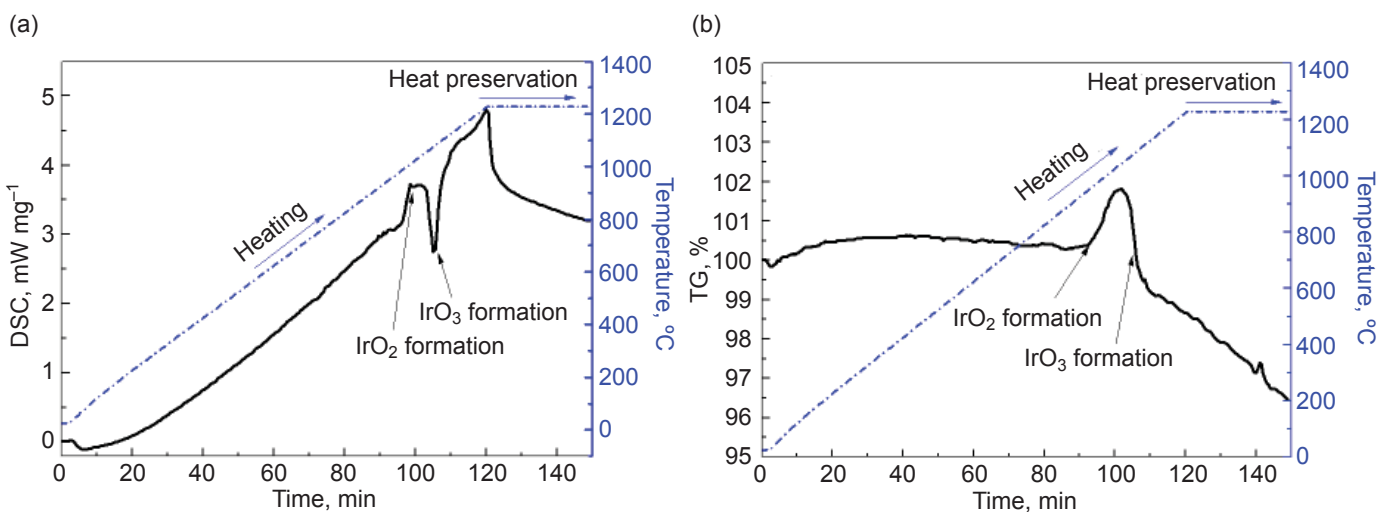


Fig. 1. (a) Differential scanning calorimetry (DSC); (b) TG lines of Ir coating (Reproduced with permission of Elsevier (6))

high-temperature environments, while a nanoporous Ir film can be used in catalytic applications due to its high specific surface area. The structure of an Ir coating is determined by the deposition processes. These include magnetron sputtering (both direct current magnetron sputtering (DCMS) and radio frequency magnetron sputtering (RFMS)) (89–91), chemical vapour deposition (CVD) (92), metal-organic CVD (MOCVD) (93, 94), atomic layer deposition (ALD) (95), physical vapour deposition (PVD), laser-induced chemical vapour deposition (LCVD) (7), electrodeposition (96–99), pulsed laser deposition (PLD) (100) and DGP (51, 101). Many processes can produce Ir coatings on various substrates, and the quality of the as-deposited coating varies. **Table II** shows the quality of Ir coatings produced by different deposition processes.

## 2.1 Slurry Dip

Slurry dip is a conventional process to prepare coatings. A single dipping operation produces a coating

and the coated specimens are heated in an argon atmosphere. The upper temperature is limited by the melting point of the coating material and this is also the limit for high-temperature applications. A strongly adherent Ir coating on graphite was obtained by fused Ir powder (111). After oxidation at 2050°C, some pores were present on the coating surface. A combination of slurry dipping and sintering, vapour-plating and electrodeposition processes could produce a dense Ir coating which could protect graphite from oxidation at 2100°C (111, 112). The slurry was made from xylene and milled Ir powder. After heat treatment, the film of Ir powder was adherent to the substrate. The adherence of the sintered Ir coating depends critically on the formation of a eutectic melt at the interface between Ir and carbon.

## 2.2 Metal-Organic Chemical Vapour Deposition

MOCVD is an advanced method of coating preparation which relies on chemical reactions of a vapour at a

**Table II Quality of Iridium Coating by Different Deposition Processes**

Method	Coating thickness, $\mu\text{m}$	Deposition rate, $\mu\text{m s}^{-1}$	Deposition temperature, $^{\circ}\text{C}$	Coating quality	Ref.
LCVD	100	–	300~400	Microcracks and non-uniform thickness	(7)
DCMS	4	$0.02 \times 10^{-3}$	25	Pores and rough surface	(55)
RFMS	4.5	$1 \times 10^{-3}$	25~800	Fine, dense, uniform and thorough coverage	(55)
Electrodeposition	50~100	$2.8\text{--}5.6 \times 10^{-3}$	~570	Large inner stress, pores and high deposition rate	(98, 102)
CVD	10	$0.28\text{--}0.56 \times 10^{-3}$	700~800	Dense and low deposition rate	(103–106)
MOCVD	5~50	$0.05\text{--}6.94 \times 10^{-3}$	500~600	Small grains and some pinholes	(13, 92, 107, 108)
PLD	~0.1	–	25~400	Well crystallised, smooth surface and low deposition rate	(100, 109)
DGP	5~50	$0.56\text{--}5.6 \times 10^{-3}$	800~1100	High deposition rate, strong bond and pinholes	(6, 12, 48, 51, 110)
ALD	0.009–0.175	0.02~0.06 nm per cycle	165~400	Low deposition rate, smooth surface, adhered well to the different substrate	(95)

surface to form solid deposits. Dense and adherent coatings depend on optimal deposition parameters and the choice of gaseous compound of the deposited material. The deposition parameters include temperature, gas concentration, carrier and co-reagent gas flow rate, precursor flow rate and gas pressure. Dense Ir coatings are essential to offer effective corrosion or oxidation protection. For generalised perturbation method (GPM) coatings by MOCVD, most of the precursors are metal organic complexes. Garcia and Goto (103) reviewed MOCVD operating conditions for Ir with different precursors such as metal  $\beta$ -diketonates, carbonyl complexes and allyl complexes. Ir coatings obtained by this route may contain carbon impurities from the thermal decomposition of organic precursors. It is very important to eliminate these carbon impurities for high-temperature applications. However, carbon can prevent the grain growth of noble metals, which leads to the formation of nanoparticles that may be highly catalytic and reversible as electrodes for solid electrolytes (113).

Some researchers (114, 115) carried out doping with precursor in the presence of oxygen to obtain pure Ir coatings by MOCVD. For the CVD process, halides of Ir as the precursors have poor volatility, the deposition temperatures are high and the reaction products will corrode the equipment (116). At present, Ir coatings made by MOCVD are successfully used in liquid rocket motors operated at  $\sim 2200^\circ\text{C}$  (45). Compared with other processes, Reed, Biaglow and Schneider (117) claimed that MOCVD is the only established process for the fabrication of Ir-coated Re combustion chambers. However, major concerns of this process are high cost, low deposition rate and impurities in the coating.

### 2.3 Atomic Layer Deposition

ALD is a superior method for thin film deposition which is used for fabrication of highly uniform and conformal Ir films over large and complex substrate areas. ALD is considered to be a special modification of CVD in which the substrate is exposed to one precursor at a time, the precursor pulses being separated by inert gas purging (95). In ALD processing, two or more precursors alternately react with the surface and saturate it with chemisorbed species. This self-limiting mechanism can lead to successful deposition of uniform and conformal films with the desired composition and thickness, as long as appropriate precursors are chosen, suitable reaction temperatures applied and the correct number of deposition cycles performed. For the deposition

of Ir, oxygen may be applied as the other precursor together with the metal complex. Molecular oxygen is activated through its dissociative chemisorption producing reactive atomic oxygen on the metal surface. The Ir complex-oxygen ALD process gives the metal only above a certain threshold temperature, which is governed most likely by the dissociative chemisorption of oxygen on the metal surface. When the deposition temperature is below this threshold, no reaction occurs and nothing is deposited (118, 119).

Hämäläinen *et al.* (120–122) reported that Ir films could be obtained using molecular hydrogen as a reducing agent in each ALD cycle after the ozone pulse at low deposition temperatures. Aaltonen *et al.* (95) observed that an Ir film with a preferred (111) orientation was grown by ALD in a wide temperature range of  $225\text{--}375^\circ\text{C}$  from tris(2,4-pentanedionato)iridium  $[\text{Ir}(\text{acac})_3]$  and oxygen. Christensen and Elam (123) synthesised Ir-Pt films using Ir(III)acetylacetonate- $\text{O}_2$  cycles for Ir ALD and  $\text{MeCpPtMe}_3\text{--O}_2$  cycles for Pt ALD at  $300^\circ\text{C}$ , and found that the growth rates of Pt and Ir remained constant regardless of the dosing ratio, indicating that both Ir and Pt ALD proceeded equally well on either metal surface.

### 2.4 Physical Vapour Deposition

PVD is a versatile technique and is the most widely used sputtering method. The sputtering process involves the bombardment of a solid target of the material to be coated with ions extracted from a concentrated plasma cloud positioned very close to the surface of the target (89, 124, 125). High energy atoms are dislodged from the target and directed toward the substrate. The advantages of PVD include a high sputtering rate at the target, high deposition rate and superior adhesion of the sputtered coating. Mumtaz *et al.* (90, 91) found that RFMS could obtain a uniform and thorough coverage of Ir with columnar grains. However, a DCMS Ir coating had a porous columnar structure and a rough surface due to the effect of shadowing. The effects of deposition parameters on the structure and properties of the Ir coating have been studied (126).

The PVD process includes electron beam PVD and plasma based ion implantation. Murakami's research group (127–129) investigated Ir-based bond coatings such as Ir-Hf and Ir-Ta prepared by electron beam PVD. An Ir-Re alloy coating created using a plasma based ion implantation process could protect engineering tools from oxidation at high temperatures (130). Lee *et al.* (131) studied Ir film deposition by electron evaporation

with simultaneous bombardment by an argon ion beam and found that the charge injection capability of the Ir film was identical to bulk Ir.

## 2.5 Electrodeposition

Electrodeposition is promising as a low cost, reliable coating process. The plating of Ir from aqueous solutions has been reviewed by Jones (132). Plating of Ir from Ir chloride solutions with sulfamic acid produces deposits up to 25  $\mu\text{m}$  thick, although the deposits exhibit cracks. Plating of Ir from solution in hydrobromic acid produces crack-free deposits up to 1  $\mu\text{m}$  thick using a deposition rate of  $\sim 1 \mu\text{m h}^{-1}$ . Improved deposition efficiencies and decreased cracking size of the coating were reported for sodium hexabromoiodate(III) baths with additions of oxalic acid. While typical thicknesses of Ir plating of  $\leq 1 \mu\text{m}$  could minimise corrosion and serve for many electronic applications, thick Ir coatings are necessary for use at elevated temperature. Cohen Sagiv, Eliaz and Gileadi (133) developed suitable plating baths for electroplating of Re-Ir-Ni alloy coating and suggested a mechanism for the electrodeposition process. Wu *et al.* (134) further studied the effects of pH and deposition temperature on the chemical composition, deposition efficiencies and microstructure of Re-Ir-Ni coatings. Qian *et al.* (135–137) reported that an Ir coating could be electrodeposited from aqueous solution, composite ionic liquid and sodium chloride-potassium chloride molten salt systems. However, the quality of the Ir coating was relatively poor.

Dense and ductile Ir coatings were electrodeposited from a molten ternary eutectic of alkali metal chlorides under an argon atmosphere as an alternative to the more generally used molten cyanide, which is toxic and unstable (98). The best electrolyte for Ir coating was a non-toxic ternary eutectic molten salt of sodium chloride-potassium chloride-caesium chloride (138). However, the electrodeposition process was performed either in inert atmosphere or in chlorine, which increased the complexity of the facility. Timofeev, Baraboshkin and Saltykova (139) carried out the electrodeposition of Ir on graphite crucibles from a fused chloride electrolyte. Saltykova (140, 141) studied the effects of electrolysis conditions on the structure of Ir deposits on graphite from a ternary eutectic molten salt using both constant and reverse current. Bai's research group (142) adopted the molten electrodeposition method to obtain a laminar Ir coating and suggested that the Ir-Re coated graphite was oxidised at elevated temperatures from 1600°C to  $\sim 1900^\circ\text{C}$  in stagnated air for 1 h. Reed

and Dickerson (143) found that an electrodeposited Ir coating presented pores and poor adherence, and suggested that the integrity of the coating could be improved by densification of the Ir layer through a post-deposition treatment.

## 2.6 Double Glow Plasma

The DGP process can be looked on as a new kind of PVD technique, which could be applied to almost all solid metallic elements to realise surface alloying of the metallic substrates (144, 145). A surface alloying experiment was performed in a DGP surface alloying device in which a glow discharge process in a vacuum sputtering chamber produced low-temperature plasma. The general advantages of this process include low operating cost, low pollution, safety, improved stability, high deposition rate, good coating uniformity, controllability of the coating thickness and strong adhesion to complex shaped substrates (146). This process can produce co-deposited coatings with different composition ratios in a controlled manner with simple operating procedures (147–149). The main characteristics of a DGP process are high deposition temperature of 800–1200°C and resputtering during the deposition process. Chen's research group (150–155) investigated the microstructure, texture evolution, growth mechanism, mechanical properties and ablation resistance of Ir coatings produced by a DGP process on refractory materials and found that the Ir has excellent adhesion, exhibits a  $\langle 110 \rangle$  texture and presents some micropores or pinholes in the coating after high-temperature treatment. Wu *et al.* (101) studied the effects of bias voltage and gas pressure on the orientation and microstructure of Ir coatings formed by DGP and found that Ir coatings on Ti, Nb and molybdenum substrates all exhibited the preferred  $\langle 220 \rangle$  orientation under the same deposition conditions. The microstructure of the Ir coating was affected by bias voltage, gas pressure and substrate effects. The bias voltages had a significant impact on the crystal orientation of the coating. The increase of bias voltage resulted in a high substrate temperature and high deposition rate. An increase in the coating thickness can affect the microstructure and orientation of the coating.

## 2.7 Pulsed Laser Deposition

The PLD process can produce high quality films with high purity and good adherence. PLD consists of a target and a substrate in a vacuum chamber. A high

power laser is used as an external energy source to vaporise the target and to deposit a thin film on the substrate. The advantages of PLD are flexibility, fast response, energetic evaporants and congruent evaporation (156). The deposition parameters include the laser characteristics, substrate temperature, degree of vacuum and the distance between target and substrate. PLD Ir films had a polycrystalline structure with average grain size in nano-scale and the PLD process led to higher purity films than RFMS (157). The crystal orientation and surface morphology were investigated by Gong *et al.* (100, 109), and the resistivities of PLD Ir films were determined as a function of substrate temperature. Well-crystallised and single-phase Ir layers with <111> preferred orientation were obtained at low substrate temperatures of 200–300°C. Chen (158) investigated high purity Ir films deposited on Si (100) *via* PLD. A high Ir deposition rate was obtained with pulsed laser power higher than  $4.2 \times 10^9 \text{ W cm}^{-2}$ . The PLD Ir films exhibited a (110) preferentially orientated polycrystalline structure. Their average grain size increased from 30 nm to 110 nm as the deposition temperature was raised from 100°C to 600°C. With a substrate temperature of 700°C, the grain size changed to 500 nm. Ir silicide was found in the film deposited at a substrate temperature of 700°C.

## 2.8 Other Processes

The PVD method for Ir coating includes both thermal evaporation and sputtering (5, 159). Bauer *et al.* (160) reported that large area, single-crystalline Ir films on  $\text{Al}_2\text{O}_3$ , strontium titanate ( $\text{SrTiO}_3$ ) and magnesium oxide (MgO) substrates are desired for the heteroepitaxial deposition of diamond. These were obtained by e-beam evaporation. Many deposition processes have been investigated for Ir coating of components. A compact Ir coating was deposited on Re by arc ion plating (AIP) (161). The isothermal oxidation resistance of the Ir coating was retained for up to 4 h at 1800°C and up to 2 h at 2000°C, respectively. After 120 oxidation test cycles at 1950°C, the surface and cross-section of the Re-Ir remained compact and no peeling was detected.

Snell *et al.* (7) reported a novel LCVD process to produce an Ir coating, the principle of which is to use a high energy laser beam to dissociate a precursor solid or liquid on the Ir slurry layer. The production of near-net-shape parts with Ir by directed light fabrication has shown some promise (162). In this method, metal powder is transported in a stream of inert gas and fused to a surface in the focus of a high power laser beam,

to form fully fused near-net-shape components. Initial work on this process indicates that porosity originating from gases during melting and solidification is an issue. Plasma spray, vacuum plasma spray or low pressure plasma spray of Ir have been proposed as alternative methods for achieving high density coatings. While there is little published literature available on plasma spraying of Ir, it is expected to perform similarly to that of a number of refractory metals.

## 3. Conclusion

Ir is of great interest to the scientific and technical communities and could be used in several applications. There are a number of processes available to produce thin or thick Ir coatings on various substrates and the quality of the as-deposited Ir coatings vary. Each process has its unique advantages resulting in different coating textures and structures. The effects on texture and structure will be explored in Part II, along with mechanical properties, growth mechanisms and an outlook for applications of Ir coatings.

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