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THIN FILM RARE-EARTH POLONIDES

June 1969

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THIN FILM RARE-EARTH POLONIDES

By

J. H. Jarrett and D. W. Brite

Fuels and Materials Department
Chemistry and Metallurgy Division

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J. H. Jarrett and D. W. Brite

ABSTRACT

The results of an investigation of the reaction of polonium-210 vapor with rare-earth films are reported. Handling techniques and reaction conditions required for preparation of thin films of rare-earth polonides with good integrity and optimum thermal stability above 1000 °C are described. Rare-earth polonide compounds having the greatest thermal stability were obtained by reaction at 1200 °C and removal of excess polonium by distillation at 1200 °C.

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INTRODUCTION

Polonium-210 has a specific power of 144 W/g, a melting point of 254 °C, and a boiling point of 962 °C.⁽¹⁾ It reacts with a number of metals to form polonides of various stoichiometries and thermal stabilities. The rare-earth polonides are among the most thermally stable polonides reported.^(2,3) A number of rare-earth monopolonides have been prepared, with melting points reported in the 1400 to 2200 °C range. All the monopolonides investigated melted incongruently with rapid loss of polonium at or above their melting points.⁽³⁾

The reaction of polonium-210 vapor with thin films of the rare earths--gadolinium, dysprosium, and erbium-- was investigated in this work. The objective was to produce a thin film of a rare-earth polonide having good integrity and optimum thermal stability.

A solid-vapor reaction process was used as a basis for these experiments. Both reactants were sealed in a stainless steel vessel as solids, and an ever-increasing polonium vapor pressure was generated within the vessel as it was slowly heated in a furnace. The excess polonium acted as a reservoir to maintain the equilibrium polonium vapor pressure as polonium was removed from the atmosphere by reaction with the rare earth. After the rare earth was completely reacted, the excess polonium was removed (cooked-out) by distillation into an extension of the reaction vessel by withdrawing the extension from the furnace.

SUMMARY

The reaction of 3 to 10 μ thick vacuum-vapor deposited rare-earth films with polonium-210 vapor was investigated. The rare earths used were gadolinium, dysprosium, and erbium. The objective was to develop a method for preparing a thin film of a rare-earth polonide that was adherent to the refractory-metal substrate and which had good integrity and optimum stability at temperatures in excess of 1000 °C. A high conversion efficiency of rare earth to rare-earth polonide was also desired.

Numerous techniques were used before, during, and after the reaction of rare earth and polonium-210 to produce a polonide of the desired properties. Oxygen contamination of the rare-earth film or the polonium-210 before the actual rare-earth polonium reaction appeared to reduce conversion efficiencies.

A significant improvement in conversion efficiency was observed after the following process procedures were adapted:

1. Have a titanium filament, heated to about 700 °C, in place before and during the vacuum vapor deposition (VVD) of the rare earth to getter out oxygen.
2. Place a glovebag around the VVD system and purge it with an inert gas. Unload the vapor-deposited rare-earth film in the inert atmosphere into a transfer container.
3. Hydride the rare-earth film by exposing it to hydrogen for about 2 hr at 650 °C.
4. Dehydride the hydrided film by heating it in vacuum for about 2 hr at 650 °C.
5. Heat the as-received polonium-210 in a sealed stainless-steel tube containing tantalum powder for 2 hr at 1000 °C. The PoO_2 that may initially be in the system is reported to decompose at 500 °C,⁽¹⁾ and the tantalum powder reacts

with free oxygen formed because of the decomposition.

Reduce the temperature of the stainless-steel tube to 700 to 800 °C and distill the polonium into an extension tube extending out of the furnace. This distillation separates polonium from lead formed by radioactive decay of the polonium-210.

6. Load the rare-earth film and freshly distilled polonium-210 into the reaction vessel only after gettering the argon atmosphere glove-box by playing the TIG welder arc on a piece of zirconium.

Since this work was developmental in nature, the individual contribution of each of the above process improvements to reaction efficiency was not fully investigated, and all of the above precautions or operating steps may not be necessary. In fact Items 3 and 4 could be eliminated with no loss in reaction efficiency if the remaining items were strictly adhered to.

The thermal stability and integrity of the rare-earth polonide films were dependent on the reaction and polonium cook-out conditions. Improved thermal stability resulted when the rare earths were reacted and cooked-out at 1200 °C. However, at this temperature, the rare-earth films were subject to damage caused by sintering and diffusion, which caused non-uniform film thicknesses. Dysprosium polonide was less subject to this problem than gadolinium polonide, probably because of its approximately 400 °C higher melting point.

Rare-earth polonides react with oxygen,^(1,2) and appropriate care must be used when handling the rare-earth polonide film after the rare-earth, polonium-210 reaction to minimize loss of polonium by oxygen replacement.

EXPERIMENTAL

MATERIALS

The rare-earth metals used in the experimental work were obtained from Research Chemicals, Phoenix, Arizona, in the form of cast 1/4 in. diam rods, 4 to 6 in. long. The metals were produced from oxides of 99.9% purity with respect to metallic oxide impurities. The oxygen content in the rare-earth metal was not measured. The polonium-210 was obtained from Mound Laboratory, Miamisburg, Ohio. The major impurities were lead (the natural decay product of polonium-210), tellurium, and oxygen.⁽¹⁾

FACILITIES

The rare-earth film was prepared in a vacuum vapor deposition (VVD) system consisting of an 18 in. diam bell jar, a 6 in. diffusion pump, and a downward evaporation electron-beam gun with a 3 kW power supply (Figure 1). A special feed mechanism was fabricated to accommodate the 1/4 in. diam rare-earth rod. A nonradioactive glove-box facility was used for hydriding, dehydriding, and final reaction preparation of the rare-earth films. The glove-box utilized an argon purge, and the oxygen level was kept below 30 ppm as measured with a Westinghouse oxygen analyzer.

The rare-earth, polonium-210 work was done in a lead-shielded glove-box facility having an argon atmosphere (Figure 2). The following equipment was used in the glove-box system:

- Mechanical Vacuum Pump: Located outside the glove-box and used to pull a vacuum on the reaction and polonium cook-out vessels.

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FIGURE 1. Vacuum-Vapor Deposition System.

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FIGURE 2. *Glove-box for Rare-Earth Polonide Work*

- TIG Welder: Used to seal the reaction and cook-out vessels. The power supply was located external to the glove-box.
- Reaction Furnace: Initially, a 1000 °C Lindberg Type 70-I tube furnace was used; later a water-cooled Marshall tube furnace capable of 1200 °C was installed.
- Various hand tools, tubing crimpers, and cutters.
- Mettler Balance, Model H-15.

ANALYTICAL TECHNIQUES

By using the following analytical techniques, the degree of rare-earth polonium reaction, the film integrity, the reaction conditions, and the polonium-210 movement in the reaction system were determined.

X-Ray Fluorescence

The amount (wt%) of rare-earth film on the tungsten substrate was determined by X-ray fluorescence analysis. The rare earth was dissolved off of the substrate with 0.1N nitric acid; a measured amount of internal standard was added to the solution, and the sample was counted. The wt% rare earth was determined by using the original weight of the rare earth and substrate, the ratio of rare earth and internal standard counts, the amount of internal standard added, and a count ratio calibration curve prepared from known rare-earth, internal-standard solutions.

Weighing

The rare-earth film and substrate were weighed with a Mettler Model H-15 balance before and after the reaction with polonium-210. Special techniques were developed for weighing the thermally hot rare-earth polonide film and substrate after the reaction.

Metallography

The film uniformity, thickness, and adherence to its substrate material were determined by using metallographic techniques. The as-deposited rare-earth films; hydrided, or hydrided and dehydrided films; and rare-earth polonide films were evaluated by using metallography. The first known successful metallographic examination of rare-earth polonides was accomplished, and the details have been reported.⁽⁴⁾

Gamma Counting

The polonium-210 movement during the course of a reaction or distillation was followed by monitoring the activity of the 0.8 MeV polonium-210 gamma rays along the length of the reaction tube. The detector, contained in a lead-shielded collimator (Figure 3), was moved at a constant rate outside the glove-box over the reaction tube. The polonium activity thus measured was plotted as a function of distance along the reaction tube on a strip-chart recorder. The equipment consisted of a NaI detector, a Hamner preamplifier, linear amplifier, single channel analyzer, count-rate meter, and a Honeywell recorder.

Temperature Measurement

The temperature in the reaction furnace was measured with either a platinum-platinum 10% rhodium thermocouple or a chromel-alumel thermocouple placed next to the reaction tube.

Calorimetry

The amount of polonium-210 in a weighed sample was determined by calorimetry. The system consisted of identical water-filled containers, with a resistance heater in one and a properly contained polonide sample of unknown power in the other (Figure 4). The temperatures in each container were

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FIGURE 3. *Holder Mechanism for Moving Gamma Detector at a Constant Rate.*

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FIGURE 4. Calorimetry Apparatus.

matched by adjusting the voltage supplied to the resistance heater. The power required to hold the temperatures equal was measured and considered equal to the power of the unknown sample. Measurements of sample heat generation rates in this equipment were estimated to be accurate within $\pm 2\%$.

POLONIDE FORMATION

The process steps used from the vacuum vapor deposition (VVD) of the rare earth through calorimetry of the final rare-earth polonide are described below:

Vacuum Vapor Deposition

The 1/4 in. diam rare-earth rod was deposited downward on a tungsten substrate by VVD. The film thickness was measured during the deposition by a Sloan monitor. The deposited films showed a uniformity of $\pm 0.5 \mu$, and ranged from 3 to 10 μ in thickness in various runs. To getter out any oxygen in the system, a resistively-heated titanium filament was kept at about 700 °C inside the deposition chamber for several hours prior to and during deposition. A glove-bag was placed around the VVD chamber and purged with either argon or nitrogen. This minimized the possibility of allowing the freshly deposited, rare-earth film to come into contact with oxygen as it was removed from the VVD system into a purged transfer container.

Rare-Earth Activation

The rare-earth film was made more reactive by hydriding or hydriding and dehydriding before exposing it to polonium-210.⁽³⁾ Hydriding involved contacting the rare earth with hydrogen at 650 °C for 2 hr, and dehydriding was accomplished by heating the hydrided rare earth for 2 hr at 650 °C in a vacuum of 20 μ . Neither operation caused the rare-earth film to lift off of the tungsten substrate. The activated rare-earth film was placed into an inert-gas purged container and taken to the polonium glove-box.

Polonium-210 Pretreatment

The polonium-210 was received from Mound Laboratory deposited on platinum gauzes. Prior to its reaction with the rare-earth foil, it was placed with some tantalum powder in a 1 in. OD stainless-steel vessel with an open 1/4 in. thin-wall stainless-steel tube on one end. A vacuum was pulled on the vessel via the 1/4 in. tube; the tube was then crimped, cut, and TIG-welded shut, with the vacuum maintained. The vessel was heated for 2 hr at 1000 °C and cooled to 700 to 800 °C; the polonium was then distilled into the 1/4 in. thin-wall cook-out tube by extending the tubing end of the vessel outside of the furnace and allowing it to cool. The purpose of this pretreatment was to allow any $^{210}\text{PoO}_2$, which may have formed on the polonium-210, to decompose to polonium and oxygen, then to have the oxygen react with the tantalum powder. Cooling the vessel to 700-800 °C prior to distilling the polonium, reduced the vapor pressure of the ^{210}Pb formed as a decay product of ^{210}Po and thus provided a partial separation from lead during the distillation. The 1/4 in. tube containing the treated polonium was crimped and cut off of the 1 in. OD vessel.

Rare-Earth Polonium-210 Reaction

The activated rare-earth film was weighed and placed with the treated polonium-210 in a 1 in. OD stainless-steel reaction vessel having an open 1/4 in. thin-wall stainless-steel tube on one end. This vessel was the same as the polonium pretreatment vessel, and it was sealed under vacuum in a like manner and placed in the furnace. An excess of polonium-210 was used in most of the reactions, and unreacted polonium was removed from the rare-earth polonide after the reaction was completed by distilling it into the cook-out tube as described above (Polonium-210 Pretreatment Section). Polonium movement was

followed with the gamma counter. Before loading and unloading the reaction vessel, the oxygen level in the argon atmosphere glove-box was reduced by heating a piece of zirconium to about 1200 °C. It was heated by playing a TIG welder arc back and forth across its surface.

The rare-earth polonide was removed from the reaction tube and weighed by placing it in a ceramic container on the balance, which minimized the weighing inaccuracies caused by thermal effects. The polonide was then placed into a small stainless-steel container, which was carefully removed from the glove-box and put into an open-face hood via another container taped to a plastic bag-out bag. The outer container was carefully sealed, removed from the bag, and placed in the calorimetry apparatus in the open-face hood.

EXPERIMENTAL RESULTS

Thirty-six fueling runs were made with rare-earth films and polonium-210. The reaction efficiencies were quite low in the initial runs, but improvements in experimental technique and equipment resulted in higher conversion of rare earth to rare-earth polonide, and in polonides with better temperature stability. The polonide film adhered to its substrate material. A chronological summary of the results is presented in Table 1.

DISCUSSION

The experimental results shown in Table 1 indicate that the rare-earth polonium fueling efficiencies and the polonide-thermal stability properties were increased as better techniques were developed and applied. Because of the developmental nature of the work, no attempt was made to fully investigate the individual contribution of each process improvement. The conversion efficiency was highly dependent on the amount of contact

TABLE 1. Summary of Experimental Results

Rare Earth Conversion, % (a)	Rare Earth	Thermal Stability of Polonide	Treatment of Rare Earth	Treatment of Polonium-210	Reaction and Cook-Out Parameters
4	Gd,Dy,Er	Not Quantitatively Evaluated	None	None	Rx 2 hr at 850 °C Cook-out same
30	Gd,Dy,Er	Not Quantitatively Evaluated	Hydrided	None	Rx 2 hr at 850 °C Cook-out same
45	Gd,Dy,Er	Not Quantitatively Evaluated	Hydrided	Heated over Ta powder	Rx 2 hr at 850 °C Cook-out same
45	Gd,Dy,Er	Not Quantitatively Evaluated	Hydrided and Dehydrided	Heated over Ta powder	Rx 2 hr at 850 °C Cook-out same
45	Gd,Dy,Er	Not Quantitatively Evaluated	None, but used during VVD and glove-bag for VVD system	Heated over Ta powder	Rx 2 hr at 850 °C Cook-out same
40	Dy	Footnote (b)	None, but used during VVD and glove-bag for VVD system	Heated over Ta powder	Rx 1 hr at 900 °C Cook-out same, then heat treated for 37 hr at 1200 °C
50	Gd	Approx. 1/3 of Po released after 45 hr at 1200 °C in vacuum	None, but used during VVD and glove-bag for VVD system	Heated over Ta powder	Rx 1 hr at 1200 °C Cook-out 45 hr at 1200 °C
84	Gd	Barely detectable Po release after 2 hr at 1200 °C in vacuum	None, but used during VVD and glove-bag for VVD system	Heated over Ta powder	Rx 2 hr at 1200 °C Cook-out same

- a. The value used for % conversion is based on the assumption that all of the rare earth reacted forms the monopolonide.
- b. Heat treated in a sealed system by a series of five-alternate reaction and cook-out cycles at 1200 °C, during which the polonide film was at 1200 °C for a total of 37 hr. Gamma-scanning showed that approximately one half of the polonium contained in the dysprosium polonide under reaction conditions alternately cooked-out and redeposited on the dysprosium polonide as the reaction vessel was shifted back and forth between the cook-out and reaction positions. This behavior indicates a reversible change in the stoichiometry of the polonide.

the rare-earth film and the polonium-210 had with oxygen before the fueling reaction. The thermal stability of the polonide was dependent on a reasonable conversion efficiency and the reaction and cook-out conditions. The conversion efficiency and thermal stability of the polonide are discussed below.

CONVERSION EFFICIENCY

The degree of conversion of rare earth to rare-earth polonide was influenced by a number of factors, but even the slightest contact with oxygen reduced conversion. Hydrided or dehydrided rare-earth films gave increased conversion efficiencies only when their contact with oxygen before reaction with polonium was minimized. As is shown in Table 1, the hydriding or dehydriding step could be eliminated with no loss in conversion efficiency as long as oxygen contact was minimized and the polonium was heated in the presence of tantalum powder prior to the rare-earth, polonium reaction.

THERMAL STABILITY

Thermal stability (retention of polonium in vacuum at elevated temperatures) of the rare-earth polonide compounds was monitored by gamma scanning during cook-out of the excess polonium. In some cases during the reaction phase of the experiment, it appeared that the excess polonium in the system (approximately 25% more than required for formation of the monopolonide) was absorbed by the rare-earth film as the system temperature reached the 1100 to 1200 °C range. When the reactor was placed in the cook-out position, this excess polonium was slowly released. The thermal stability of the polonides that remained following this treatment was greater than that of the polonides formed at a maximum temperature of 800 to 900 °C, under which conditions the excess polonium did not deposit on the rare earth.

In the previous work referenced,⁽³⁾ formation of higher rare-earth polonides was suggested. It may be that in this work such compounds were formed at 1100 to 1200 °C and reverted to the monopolonides or other lower polonides when the polonium vapor pressure was reduced by distillation. Apparently, a more stable compound resulted from decomposition of the higher polonides than from direct reaction at a lower temperature.

Rare-earth polonide compounds completely stable at 1200 °C were not prepared although, in some cases, polonium loss rates were so low that several hours observation were required for detection of the polonium loss. It is likely that a more quantitative study of the formation and decomposition of higher polonides could lead to a further improvement in the thermal stability of the resultant rare-earth polonide compounds.

CONCLUSIONS

As a result of this work, the following conclusions were drawn:

- Oxygen contamination of the rare-earth film, resulting in low reactivity towards polonium, may take place during vacuum vapor deposition (VVD). This is indicated by deposition vacuum of less than 10^{-5} torr.
- The rare-earth film must not be exposed to oxygen before it is reacted with polonium.
- A time delay of up to 10 days does not affect the reactivity towards polonium of hydrided (2 hr, 650 °C) rare-earth films.
- For reproducible fueling efficiencies, polonium must be distilled over tantalum powder for 1 hr at 1000 °C prior to the actual fueling run.
- A 45% conversion of rare earth to rare-earth monopolonide may be obtained by hydriding or hydriding and dehydriding the rare-earth films prior to reacting them with polonium for 2 hr at 850 °C.

- Reacting rare earths with polonium at 1200 °C yields polonide compounds more thermally stable at 1200 °C than reacting and cooking-out at lower temperatures (850 °C).
- Processing rare-earth polonide films at 1200 °C causes reduced film uniformity and integrity as a result of sintering and diffusion which occur much more extensively at this temperature than at lower temperatures.

RECOMMENDATIONS

It is recommended that future work in this area include further investigations of the following subjects:

- Thermodynamic analyses of possible system reactions.
- Experimental work aimed at discovering a rare-earth polonide compound having optimum properties relative to both thermal and structural stability at the 1200 °C temperature region.
- A quantitative investigation of the stoichiometry of thermally stable rare-earth polonides and of the precise nature of their formation and decomposition reactions.

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