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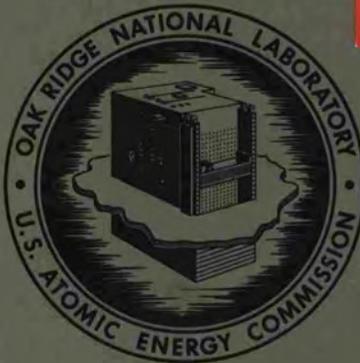
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THE STATUS OF EUROPIUM COMPOUNDS FOR REACTIVITY
CONTROL IN NUCLEAR REACTORS

R. J. Beaver and M. M. Martin

Paper presented at the Rare Earth Applications Symposium
of the American Institute of Chemical Engineers, Materials
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CONTENTS

	<u>Page</u>
Abstract	1
Introduction	1
Nuclear Properties	2
Europium and Its Compounds	3
Compound Preparation	5
Preparation of Dispersions	7
Corrosion Test Results	9
Irradiation Test Results	12
Summary	14
Acknowledgments	15



THE STATUS OF EUROPIUM COMPOUNDS FOR REACTIVITY
CONTROL IN NUCLEAR REACTORS

R. J. Beaver and M. M. Martin

ABSTRACT

Europium is an attractive element for controlling excess reactivity in thermal reactors because of its high thermal neutron absorption cross section of 3980 barns and its inherent resistance to irradiation damage. Compounds of europium, particularly europium sesquioxide (Eu_2O_3), europium titanate ($\text{Eu}_2\text{Ti}_2\text{O}_7$), and europium molybdate ($\text{Eu}_{5.3}\text{MoO}_{11}$), have been developed for these applications. Procedures are described for preparing these compounds and dispersing them in stainless steel and aluminum. Chemical stability, physical properties, and irradiation behavior of these dispersions are reviewed.

INTRODUCTION

One of the most significant achievements in the peaceful use of nuclear energy is the controlled fissioning of ^{235}U in a reactor to produce steam and, ultimately, electricity. Certain elements have the ability to strongly absorb neutrons that are produced by the fission process, and inserting such materials judiciously into a reactor "poisons" the reaction. The effect may be likened to applying the brakes in your car in that it controls the rate of the reaction. Boron, cadmium, and hafnium represent the most popular control-rod materials. However, europium in the past several years has risen from an interesting curiosity to a position of definite utility and application. The annual supply presently needed by the nuclear industry is estimated at 100 kg, and this requirement may well double by 1975. A number of reasons exist for this increasing interest in europium and its compounds, and the intent of this paper is to examine these reasons and review recent advances in europium control-rod technology.

NUCLEAR PROPERTIES

Europium principally undergoes an ${}^N\text{Eu}(n,\gamma){}^{N+1}\text{Eu}$ reaction in nuclear reactors. This reaction occurs without the formation of gaseous products; therefore, the material should be dimensionally stable under irradiation. In contrast, control rods that contain boron-bearing materials generate substantial quantities of helium from the $\text{B}(n,\alpha)\text{Li}$ reaction. This gas with the attendant swelling limits the effective life of the boron-containing rod even though only a relatively small fraction of the boron has been consumed.

Another major advantage of europium is that it retains a high thermal neutron absorption cross section over a long exposure. This effect was described by H. E. Stevens¹ in 1958 and is illustrated in Fig. 1, which shows the variation of thermal neutron absorption cross section of natural europium as a function of the number of neutrons absorbed per initial europium atom. Europium initially exhibits a

¹H. E. Stevens, Nucl. Sci. Eng. 4, 373-385 (1958).

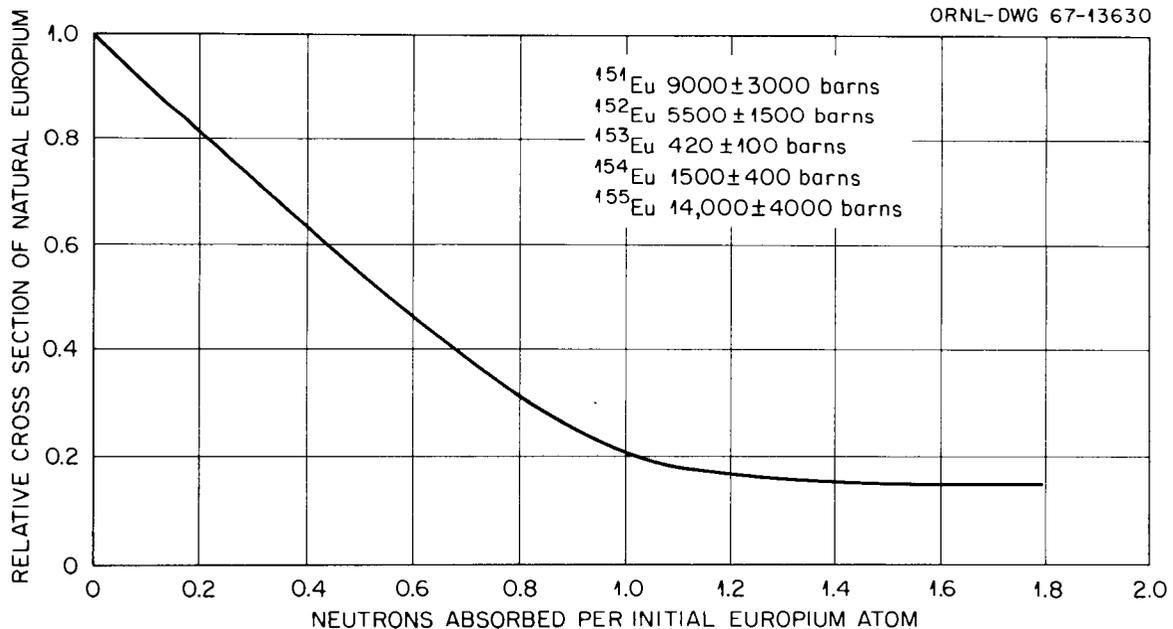
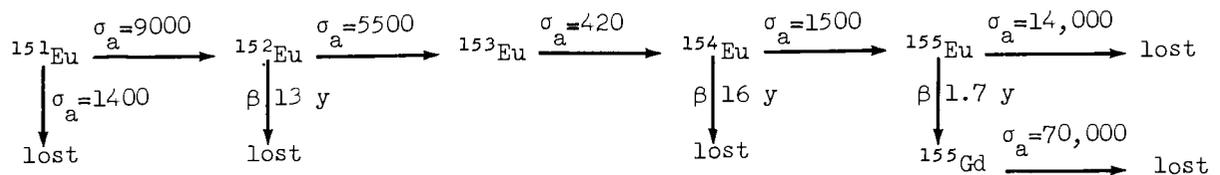


Fig. 1. Variation of the Neutron-Absorption Cross Section of Natural Europium with Absorption of Neutrons. Natural europium contains 47.8% ${}^{151}\text{Eu}$ and 52.2% ${}^{153}\text{Eu}$.

cross section of 3980 barns. As indicated in Fig. 1, a substantial cross section of 800 barns remains after the absorption of one neutron per initial europium atom. The reason for this phenomenon is primarily the retention and growth of the 420-barn ^{153}Eu combined with the birth and growth of the 1500-barn ^{154}Eu and 14,000-barn ^{155}Eu , as shown in the diagram:



Therefore, the effective lifetime of europium-bearing control rods can be appreciable.

EUROPIUM AND ITS COMPOUNDS

Europium is a highly reactive metal when exposed to oxygen or moisture and thus has found little application as pure material. At present the utility of europium is restricted to specific compounds that can be produced and handled safely and relatively economically. Europium sesquioxide (Eu_2O_3), titanate ($\text{Eu}_2\text{Ti}_2\text{O}_7$), and molybdate ($\text{Eu}_{5.3}\text{MoO}_{11}$) are the compounds that have received considerable attention. Pertinent properties of these compounds are shown in Table 1.

When prepared by the usual oxalate precipitation process, Eu_2O_3 is body-centered cubic at room temperature. However, on heating to between 1000 and 1100°C it transforms, apparently irreversibly, to the monoclinic form. Europium sesquioxide offers the highest europium density of all of the compounds listed — a distinct advantage in fabrication where high concentrations of europium are desired in a dispersion. Unfortunately, this monoclinic sesquioxide, like the metal, is unstable in the presence of moisture, forming an extraneous phase postulated to

Table 1. Pertinent Characteristics of Europium Compounds

Compound	Density (g/cm ³)	Crystal Structure	Europium Content (wt %)	Europium Density (g/cm ³)
Eu ₂ O ₃	7.99	Monoclinic	86.4	6.9
	7.28	bcc		
Eu _{5.3} MoO ₁₁	7.20	fcc	74.7	5.4
	7.17	Rhombohedral		
Eu ₂ Ti ₂ O ₇	6.01	Pseudocubic	59.4	3.6

be a hydrated oxycarbonate.² This phase is believed to be the cause of instability of Eu₂O₃ when it is subsequently exposed to the hydrogen sintering treatments used in the preparation of Eu₂O₃-stainless steel compacts.

Because of the hydration problem with Eu₂O₃, the titanate³ and molybdate⁴ were developed in an effort to produce a corrosion-resistant europium compound. These compounds have the formulas and properties shown in Table 1 and have been prepared generally by solid-state reaction of the constituent oxides at high temperatures. The titanate exhibits only one crystal structure: a pseudocubic. The molybdate is rhombohedral at low temperature but transforms to face-centered cubic above 1500°C. The cubic form can be retained by rapid cooling. Since the molybdate has a 50% higher europium density than the titanate, it is more attractive for certain applications. Both compounds are resistant to corrosion by water.

²R. A. McNees and R. A. Potter, Army Reactors Program Progress Report, ORNL-3231 (January 1962), pp. 16-22.

³W. Precht and H. Barr, Investigation of Stabilized and Hydration Resistant Europium Oxide, MND-M-2346 (June 1960).

⁴R. A. Potter, R. E. McDonald, and C. F. Leitten, Jr., "Preparation and Properties of Lanthanum Molybdates and Tungstates," pp. 547-556 in International Symposium on Compounds of Interest in Nuclear Reactor Technology, ed. by J. T. Waber, P. Chiotti, and W. N. Miner, AIME Metallurgical Society, New York, 1964.

COMPOUND PREPARATION

All of these compounds can be considered for reactor application in their bulk form, either as vibratorily compacted powder or as pressed-and-sintered pellets. However, their use has generally been confined to dispersions of the compound in a metallic matrix.

When europium compounds are to be mixed with metallic powders subsequently densified by rolling or extrusion, the dispersoid should consist of highly dense entities to avoid fragmentation and stringering. The low-density, finely divided oxide produced by calcination of europium oxalate and typified by the specifications listed below must therefore be densified by sintering at an elevated temperature.

Eu ₂ O ₃ content, wt % (min)	98
Crystal structure	bcc
Silicon content, ppm (max)	200
Ignition loss, wt %	< 0.1
Alpha activity, dpm/mg sample (above background)	10

The Eu₂O₃ is densified by pressing the powder into pellets and sintering in hydrogen at 1700°C. This treatment yields the monoclinic modification. Compacts of titanate and molybdate can be prepared similarly.

A significant advance in the manufacturing of dense europium compounds was made at ORNL. To produce production-scale quantities of dense material, an inert-gas arc-fusion technique was developed. This practice was first used to prepare europium molybdate and titanate and extended to dense europium sesquioxide. A schematic drawing of the arc-melting furnace is shown in Fig. 2. It uses a hollow thoriated-tungsten electrode tip and a water-cooled copper melting hearth. Arc instability encountered at first during melting was caused by gas evolution from the dissociation of hydroxides and carbonates. Passing the inert gas mixture through the hollow tungsten electrode stabilizes the arc by sweeping the evolving contaminants from the immediate vicinity of the arc. A deep hemispherical mold cavity reduces arc scatter associated with spreading of small droplets of the molten material. Batches

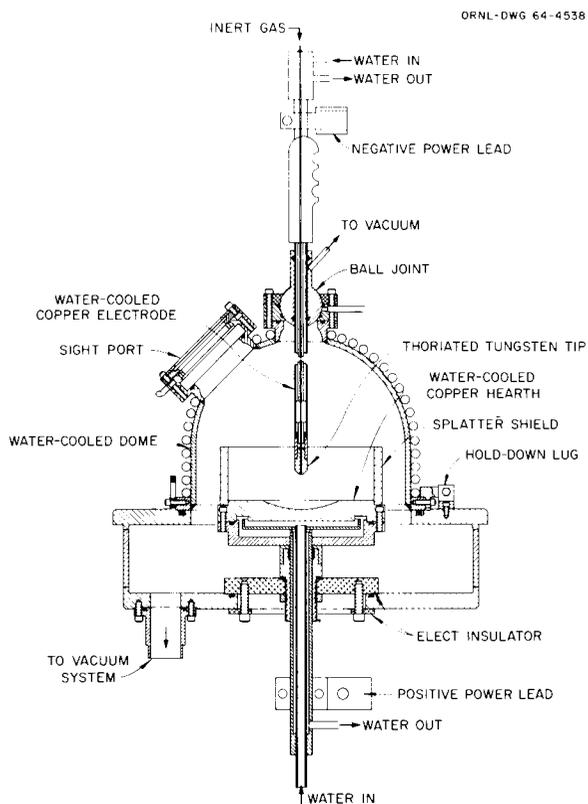


Fig. 2. Laboratory-Size Inert-Gas Nonconsumable Arc-Melting Furnace.

of 300 to 500 g are normally fused in two steps. First, low-density cold-pressed pellets are partially fused to remove nearly all of the volatile components. Then the material is melted until the powder is completely fused. During this stage, the power is kept low (300 to 400 amp at 30 v) until the oxide charge melts; then it is increased gradually to 800 amp at 40 v to ensure complete fusion of the oxide.

The inert-atmosphere arc fusion of the europium sesquioxide, molybdate, and titanate always yields dense, vitreous-appearing products. In contrast to the gray color of the air-fused compounds, the darkened color of the arc-fused material indicates an oxygen deficiency. Heating the arc-fused compounds in air at approximately 1000°C readily restores the oxygen content. These compounds are easily crushed to the particle size ranges of interest by conventional disk grinding with intermediate screenings to minimize the generation of excessive fines. Cylindrical shapes of these materials can also be readily obtained by arc drop

casting. Cylinders up to 4 in. in length and 1 in. in diameter have been successfully produced.

Preparation of these materials and their fabrication into and use as dispersions in stainless steel have been reviewed.⁵

PREPARATION OF DISPERSIONS

Conventional fabrication practices can be readily used to prepare dispersions of the described compounds in stainless steel and manufacture of clad components by hot rolling when the concentration of the europium compound is restricted to less than 40 vol %. Figure 3 illustrates the characteristics of a hot-rolled stainless-steel-base produce containing 36 wt % Eu_2O_3 . The homogeneity of the -325 mesh dispersoid is excellent. In this product, a density of 95% was achieved by the combination of sintering the pressed Eu_2O_3 -stainless steel blend for 75 min at 1200°C in hydrogen and coining at 62,000 psi at room temperature. The densified core was clad with stainless steel by rolling at 1150°C to a 90% reduction in thickness.

Both europium sesquioxide and molybdate have been successfully incorporated into aluminum-base plates by powder metallurgical techniques and cladding by hot rolling. Figure 4 illustrates the structure of a rolled aluminum dispersion containing 59 wt % -100 mesh Eu_2O_3 particles. To prepare the dispersion, the powder was blended and then compacted to 90% of theoretical density by pressing under 45,000 psi at room temperature. After degassing at 590°C to remove pressing lubricants, the compacts were clad with aluminum by hot rolling at 500°C to an 88% reduction in thickness.⁶

To avoid reaction of europium sesquioxide and molybdate (but not titanate) with the metallic matrix and undesirable dimensional instability,

⁵C. F. Leitten, Jr., and R. J. Beaver, "Use of Lanthanide Oxide Neutron Absorbers in Pressurized Water Reactors," Nucl. Appl. 4, 399-417 (1968).

⁶R. J. Beaver, A. E. Richt, and M. M. Martin, Irradiation Behavior of Aluminum-Base Dispersions Containing Europium Oxides, ORNL-4199 (January 1968).

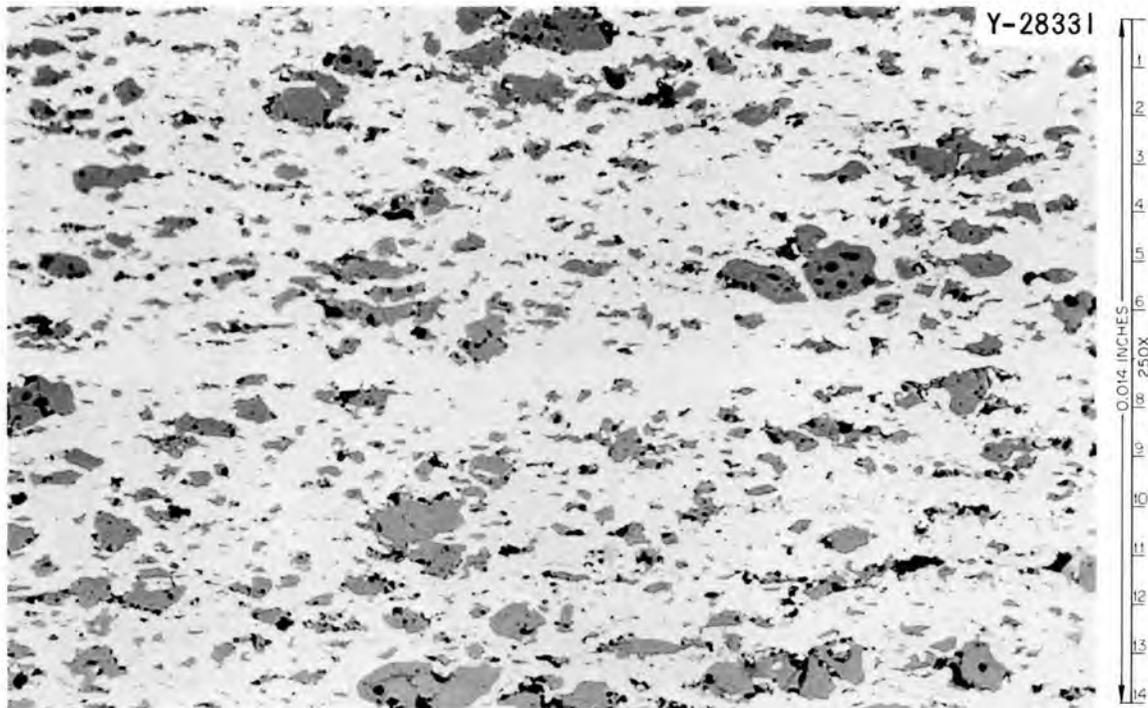


Fig. 3. Typical Microstructure of Fabricated Stainless-Steel-Clad Plate Containing 36 wt % Eu_2O_3 . As polished.

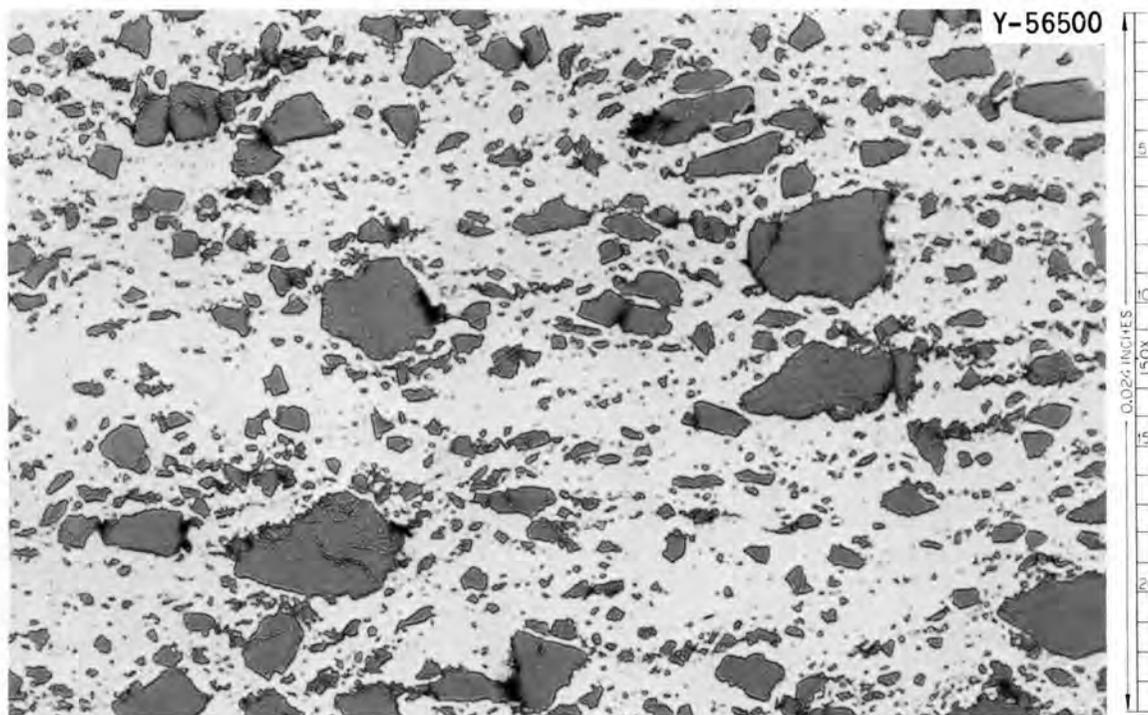


Fig. 4. Typical Microstructure of Fabricated Aluminum-Clad Plate Containing 59 wt % Eu_2O_3 . As polished.

the silicon in the matrix⁷ should be limited to less than 0.03 wt %. The evidence of this reaction in a pellet sintered at 1230°C containing 30 wt % Eu_2O_3 in type 347B stainless steel is shown in Fig. 5. After being sintered 1.5 hr at 1230°C, the pellet has increased 6.8% in volume, and the "halo-type" reaction products surrounding the stainless steel particles are clearly visible. Although the reaction zone is multiphase, EuSi_2 was the only compound positively identified. Also reaction of Eu_2O_3 with silicon produces catastrophic failures, but the iron, nickel, and chromium ingredients of stainless steel do not react when the silicon content is less than 0.03 wt %. Under similar testing conditions europium titanate does not react, and silicon-bearing stainless steels are acceptable with this dispersoid.^{5,8}

Since ceramic particles are usually brittle and of low thermal conductivity, their dispersions in a metallic matrix are generally significantly lower in ductility and thermal conductivity than the matrix material. Europium sesquioxide is no exception. As shown in Table 2, a 59 wt % Eu_2O_3 dispersion in aluminum has substantial strength, but its ductility has been drastically reduced.⁹ This addition of europium oxide reduced the thermal conductivity to approximately one-third of that for aluminum.

CORROSION TEST RESULTS

Although components containing the europium compound are ordinarily clad, the possibility of an inadvertent defect makes desirable that the compound itself be corrosion resistant to the reactor water coolant.

⁷C. F. Leitten, Jr., The Stability of Europium Oxide in Silicon-Bearing Stainless Steel, ORNL-2946 (Aug. 6, 1960).

⁸C. F. Leitten, Jr., "Nuclear Poisons and Control Applications of Ceramic Materials," pp. 435-451 in Proceedings of the Conference on Nuclear Applications of Nonfissionable Ceramics, Washington, D.C., May 9-11, 1966, American Nuclear Society, Hinsdale, Illinois, June 1966.

⁹R. J. Beaver, C. F. Leitten, Jr., and G. M. Adamson, "The Development of Aluminum-Base Eu_2O_3 Control Rods for the High-Flux Isotope Reactor," (summary) Trans. Am. Nucl. Soc. 10(1), 137-138 (June 1967).

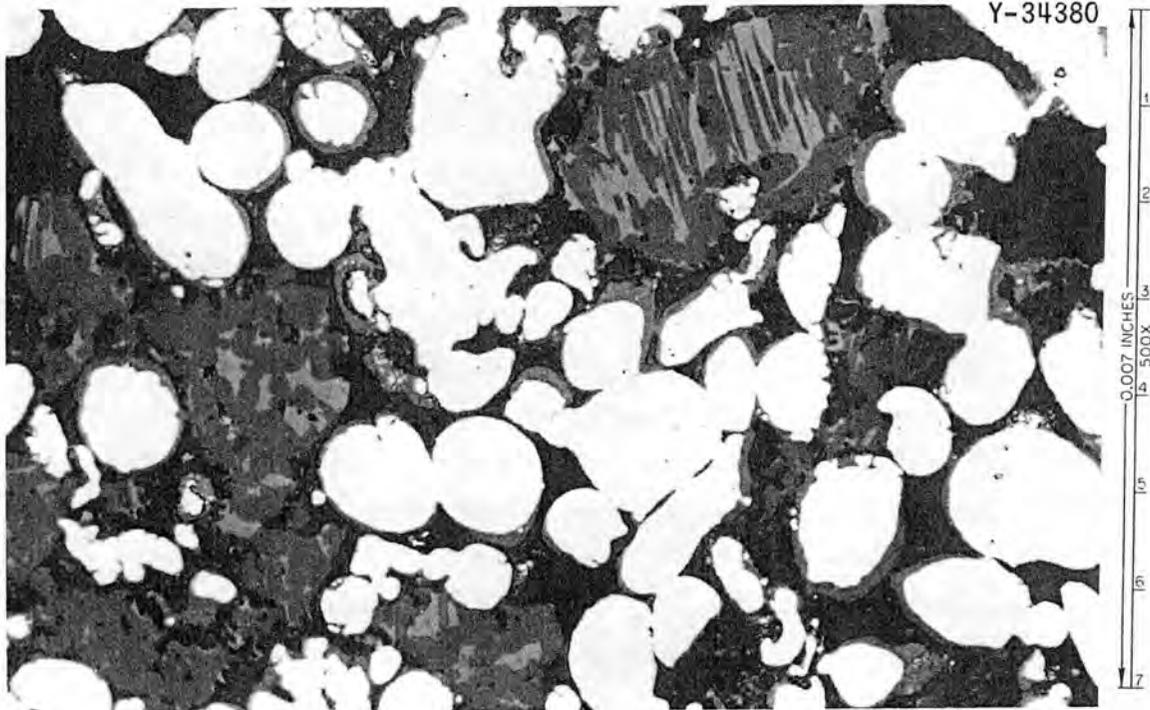


Fig. 5. Microstructure of a 30 wt % Eu_2O_3 Dispersion in Type 347B Stainless Steel Heat Treated at 1230°C for 1.5 hr Under Hydrogen. As polished.

Table 2. Pertinent Properties of Alclad Plate Containing 59 wt % Eu_2O_3 Dispersed in Aluminum

Tensile strength, psi	
Parallel to rolling direction	11,800
Transverse to rolling direction	10,300
Elongation, %	
Parallel to rolling direction	5
Transverse to rolling direction	3
Thermal conductivity, ^a watts meter ⁻¹ °C ⁻¹	
At 31°C	80.2
At 71°C	85.8

^aAluminum: 220 watts meter⁻¹ °C⁻¹ at 0 to 100°C.

Figure 6 is a striking example of the nature of the hydration of Eu_2O_3 when exposed to water at 100°C . The specimen illustrated is a rolled aluminum-clad product containing 59 wt % Eu_2O_3 . As a deliberate defect, a 0.030-in. hole was drilled through the cladding into the core, and the specimen was exposed for 1 hr to water at 100°C . Water has penetrated extensively and the Eu_2O_3 has hydrated with attendant swelling, causing

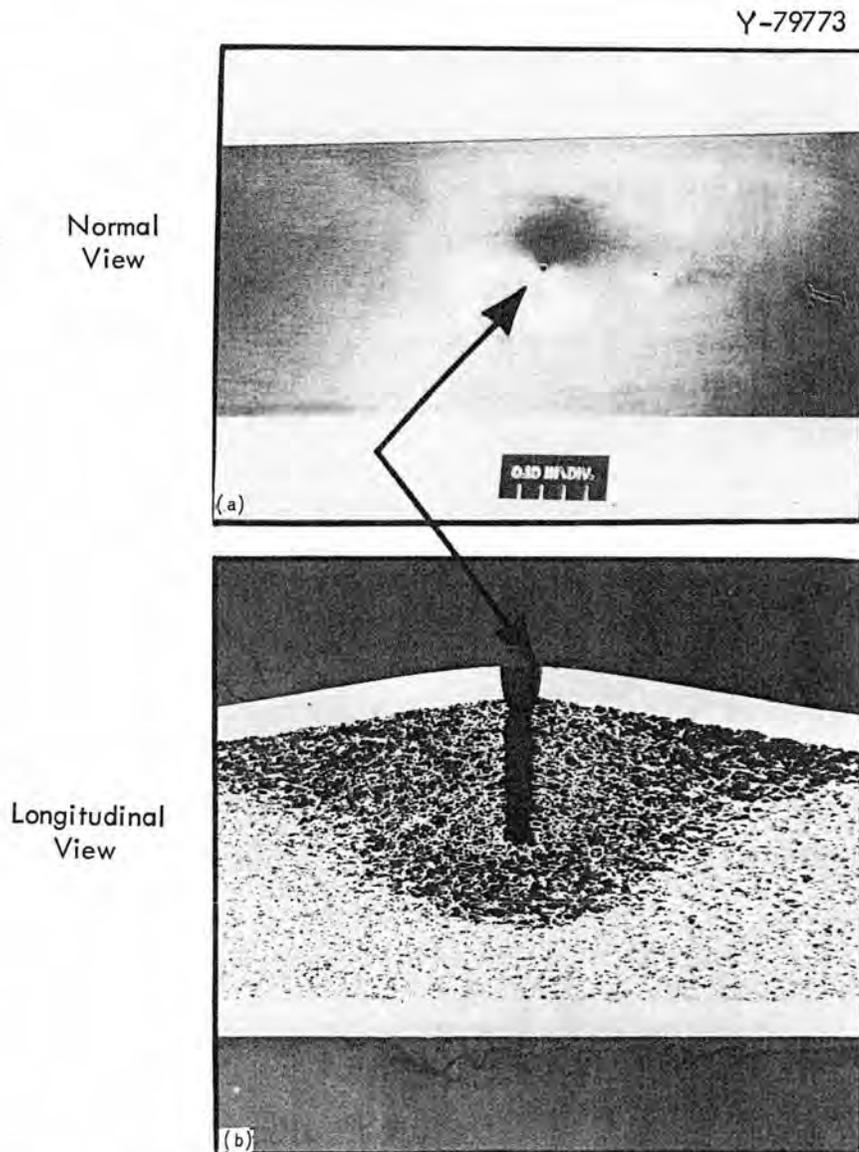


Fig. 6. Corrosion Effects in Intentionally Defected Absorber Plate After 100-hr Exposure to Water at 100°C . (a) Normal view showing swollen region at area of defect. (b) Longitudinal cross section showing extent of attack to the Al-33 vol % Eu_2O_3 dispersion. 7 \times . Reduced 35%.

the localized blister. Figure 7 shows the results of exposing 20 and 40 wt % Eu_2O_3 dispersions in stainless steel for 88 hr to 300°C pressurized water.¹⁰ The water penetrated extensively into the 40 wt % Eu_2O_3 specimen. The resultant hydration of the Eu_2O_3 created severe swelling and caused the dispersion to exfoliate. In comparison, negligible penetration into the specimen containing 20 wt % Eu_2O_3 occurred because sufficient stainless steel matrix was available to essentially surround each Eu_2O_3 particle. The additional stainless steel thus prevented the formation of an interconnected path by which water could reach interior Eu_2O_3 particles.

Corrosion tests in water at 100°C for 140 hr on europium titanate¹¹ and for 1250 hr on the molybdate⁸ failed to show any significant weight change. In pressurized water at 250°C the weight change of the titanate was merely 0.04% for a 140-hr test. The molybdate after an 850-hr test in a similar environment showed a 4% weight change. In all tests the europium compounds remained physically intact and showed no visible signs of degradation.

IRRADIATION TEST RESULTS

The stainless-steel- and aluminum-base plate-type specimens containing europium sesquioxide and molybdate exhibited acceptable dimensional stability after extensive exposure to thermal neutrons,^{10,12,13} as shown in Table 3. The thickness change is an excellent monitor of the dimensional stability of plate specimens and, in all specimens containing europium sesquioxide, was less than 0.5%. Such small increases

¹⁰C. F. Leitten, Jr., A. E. Richt, and R. J. Beaver, Irradiation Behavior of Eu_2O_3 -Stainless Steel Dispersions, ORNL-4104 (June 1967).

¹¹W. Precht and H. Barr, Investigation of Stabilized and Hydration Resistant Europium Oxide, MND-M-2346 (June 1960).

¹²R. J. Beaver, A. E. Richt, and M. M. Martin, Irradiation Behavior of Aluminum-Base Dispersions Containing Europium Oxides, ORNL-4199 (January 1968).

¹³C. F. Leitten, Jr., and R. J. Beaver, "Use of Lanthanide Oxide Neutron Absorbers in Pressurized Water Reactors," Nucl Appl. 4, 399-417 (1968).

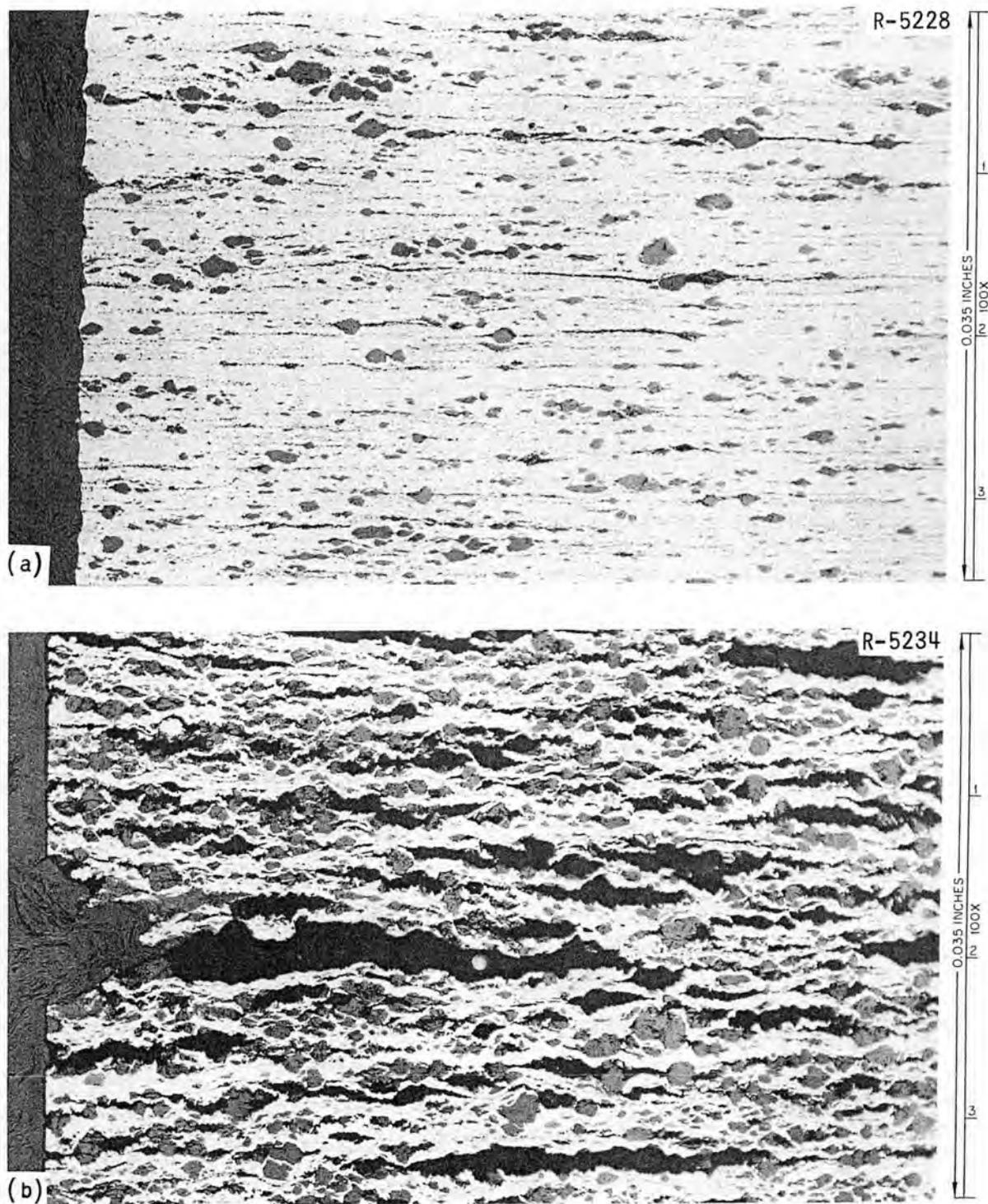


Fig. 7. Typical Microstructures of Eu_2O_3 Dispersions in Stainless Steel After 88 hr Exposure at 300°C to Pressurized Water. As polished. (a) 20 wt % Eu_2O_3 . (b) 40 wt % Eu_2O_3 .

Table 3. Irradiation Test Results on Stainless Steel- and Aluminum-Base Plate Specimens Containing Europium Sesquioxide and Molybdate

Type of Dispersion	Thermal Neutron Absorptions (neutrons/cm ³)	Increase in Thickness (%)
	$\times 10^{20}$	
20 wt % Eu ₂ O ₃ in type 304 stainless steel ^a	26	0.2
	41	0.3
30 wt % Eu ₂ O ₃ in type 304 stainless steel	18	0.2
	57	0.1
40 wt % Eu ₂ O ₃ in type 304 stainless steel	40	0.2
	64	0.2
59 wt % Eu ₂ O ₃ in type 101 aluminum	16	0.2
	41	0.5
65 wt % Eu _{5.3} MoO ₁₁ in type 101 aluminum	29	0.9

^aSilicon content in stainless steel < 0.03 wt %.

are remarkable because the "high burnup" specimens were exposed to a neutron flux of 1×10^{14} neutrons cm⁻² sec⁻¹ for 15 to 18 months. Essentially all of the thickness increase in the specimen containing the europium molybdate occurred during the first six weeks. The magnitude of this increase is not considered unacceptable but, in comparison to the Eu₂O₃-aluminum tests, is a surprising result and not clearly understood. Metallographic examination of the microstructures of these specimens showed that both europium compounds were completely compatible with both the stainless steel and aluminum matrices.

Dispersions of europium titanate have not been irradiation tested. However, a 55 wt % dispersion of this compound in type 304 stainless steel is being used in the control rod of the PM-3A reactor in Antarctica.

SUMMARY

Europium sesquioxide and its corrosion resistant counterpart, europium titanate, are being used extensively to control the excess

reactivity in operating power reactors. Europium molybdate appears to be a promising addition to the family of compounds that resist corrosion in water. These compounds can be readily prepared in a highly dense form by arc fusion. Components containing dispersions of these compounds in stainless steel and aluminum can be produced by conventional powder metallurgical techniques and hot-rolling operations. To ensure chemical and dimensional compatibility in stainless-steel-base dispersions containing europium sesquioxide and europium molybdate, the silicon in the base metal should be restricted to less than 0.03 wt. %.

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