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REFINING AND PURIFICATION OF RARE-EARTH METALS

by

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REFINING AND PURIFICATION OF RARE-EARTH METALS

Introduction

The rare earth metals currently available, whether produced metallothermically or by electrolytic means, may generally be characterized as being of no better than "commercial" purity. Most have been prepared with 1% to 2% total impurities, and a number of producers will guarantee this level (1). By scrupulous control of processing variables and raw material purity, particularly in the metallothermic reduction process, most of the metals can be prepared with 0.5% or less total impurities. Such processing care greatly increases the cost of an already expensive product, however, and thus far few of the metals have had sufficient purity requirements to justify the added cost.

In the case of yttrium, relatively high purity was found to be a requirement for workability, and it may be presumed that this will also be the case with other members of the group as uses develop which require metal fabricability. Of more immediate importance, however, is the full characterization of the metals through accurate determination of their properties. The need for high purity is paramount here, but relatively small quantities of metal are required, and as a consequence, it may be more feasible to consider means of beneficiating a relatively impure metal than to attempt ultra refinement of the reduction technique.

Advances in the field of "reactive" metals and semiconductors, where rigid control of impurities is vital, have prompted the development in recent years of a number of techniques of metal purification, all of which employ an impure metal rather than a compound as the starting material. Among these are zone refining, vacuum processing, deposition by the van Arkel - de Boer process, and solid state electrolysis. In general, purification of only a few of the rare earth metals has been attempted by any one of these techniques, but these trials give at least some indication of what may be expected with other members of the group.

Nature of Impurities

Before discussion of the purification processes themselves, it is advisable to discuss briefly the nature of the impurities which are found in the rare earth metals. These, of course, are dependent on the source and purity of the raw materials used in the reduction, and the nature of the reduction itself.

In general, present-day separation techniques make feasible the isolation of a single member of the rare earth group from its neighbors to any desired degree. Thus the remaining rare earth impurities in a "commercial" metal of the group would not normally exceed 0.1%. None of the purification techniques to be described is particularly effective in removing these impurities, and they will, therefore, remain essentially unchanged in the purified metal.

Of much greater importance are the other metallic impurities which may be found in the metals as initially produced. Principal offenders in metallothermic reduction products will be the reductant and any metal added to the charge for producing a low melting alloy. In the usual case these will be calcium and magnesium respectively, though lithium and zinc have been used. Despite the subsequent vacuum treatment for removal of the alloy constituent, as much as 1% of these metals may remain in the sponge. Consequently, the processing of the commerical metals is almost always completed by melting and casting under the highest possible vacuum. By this means, the Ca and Mg content can be lowered to a maximum of 150 ppm each (2), and may be further reduced to a considerable extent if multiple melting is employed.

Other metallic impurities result from attack on containers in various stages of preparation of the raw materials, or in the reduction itself. These include iron, tantalum, titanium and zirconium, though all are not generally associated as major impurities with one process or product.

When the reduction is carried out in tantalum, the level of this contaminant averages 0.05% in the resulting metal, but may be much higher if long times or high temperatures are employed. When an alloying element is added to lower the melting point, as in the case of magnesium in the yttrium reduction, titanium and zirconium may be used as reaction vessels. In comparable reductions of yttrium, titanium pickup averaged 0.15% while that of zirconium averaged 0.58%.

In the electrolytic reduction, extensive corrosion of the cell may occur and products of this process may be very highly contaminated with iron, carbon, silicon, and other metallic impurities.

Probably of greater significance in their effects on properties of the metals are the nonmetallic and interstitial impurities, oxygen, nitrogen, carbon, hydrogen, and the halogens. Though little study of the systems of these elements with the metals has been made, it is belived that they generally exhibit low solubilities (at least at the lower temperatures), and consequently are present in the metals primarily as compound inclusions, e.g. oxides, nitrides, halides, oxyhalides, etc. Their presence results from contact of the reactants or the products with impure atmospheres or from incomplete reactions or separations in initial preparation steps. Since they generally appear as compounds, their volume concentration in the metal is great and their effect on metal properties extensive. In yttrium, for example, oxygen content of about 0.3% and fluorine content of 0.08% result in approximately 5 volume per cent of inclusions as estimated from metallographic examination.

In Table I is shown a fairly complete and typical analysis of routinely produced yttrium metal as prepared by the Ames process of fluoride reduction with calcium and magnesium in a zirconium reaction vessel followed by vacuum "demagging" and double arc melting by the consumable electrode process. While the yttrium reduction is more difficult than some others and purity may therefore be somewhat lower than the average, this material is probably not too dissimilar in analysis from other metallothermically reduced rare earth metals. Figure I shows the microstructure of this material. The many inclusions are evident, and the structure can only be described as "dirty".

Determination of Purity

The analysis of impurities in the rare earth metals has been the subject of some study (3, 4). There are no clear-cut and undisputed techniques, however, and this is particularly true for low impurity levels. For direct chemical analysis, the most widely used techniques are spectrographic determination of the metallic elements, vacuum fusion for O, N, and H, combustion in oxygen for carbon, and pyro-hydrolysis for the halogens. An assay for total rare earth content is usually performed by oxalate precipitation.

In each case, there are existent but ill defined lower limits of sensitivity, and several of the impurities fall below these limits, even in the "commercial" metals. When purity is increased to the 99.9% level and higher, it is evident that only the most abundant impurities will be detectable, and these with questionable quantitative accuracy.

Alternate analytical techniques have been developed for some impurity elements, as for example, the spectroscopic technique of Fassel and coworkers (5) for oxygen, but these do not in general extend the sensitivity limits to any significant degree. Of possibly greater value is the technique of radioactivation analysis (6,7), which, though complex and expensive, offers the possibility of detection of exceedingly small amounts of impurities. This technique has thus far only been employed for determination of specific rare earths as impurities in a single member of the group, however.

Consequently, as purity is increased, its measurement becomes less quantitative and more a matter of indirect surmise from observed properties. One very important area is metallographic examination. Since, as noted earlier, many non-metallic impurities occur as compound inclusions, a fair idea of purity may be obtained from the cleanliness of the microstructure. The techniques for metallographic specimen preparation have been described for the rare earth metals (8).

Other properties commonly used for estimation of purity are hardness, workability, and electrical conductivity. These are of comparative value only, however, and in some cases are quite sensitive to structure and thermal history.

Zone Refining

The technique of zone refining is of very recent origin, and has been the key to successful production of ultra-pure metals for semiconductor use. The technique has been comprehensively described by Pfann, its discoverer (9). The principle is simply that most impurities have a different equilibrium solubility in the solid and liquid phases of a freezing system, and that a transfer of the impurity can consequently be obtained by passage of a short molten zone through a relatively long solid section. Obviously the determination of the ultimate purification obtainable in this way requires knowledge of the phase diagram for the impurity - metal system, particularly in the low impurity concentration range. Such data are rarely available, and this is particularly true in the case of the rare earths. Thus, the determination of feasibility of zone refining for a particular metal is usually approached experimentally.

Two basic techniques are employed for the passage of molten zones. In the simpler case, the metal is contained in a long trough or crucible and the zone is passed in a horizontal direction. Many metals, and many of the rare earth metals in particular, are so reactive that they reduce ceramic container materials and interdiffuse with metallic containers at their melting points with subsequent contamination. Some attempts have been made to overcome this problem by use of a water cooled container in which an unmolten skin of the metal being purified is maintained and acts as the true container of the molten metal. Of greater importance, however, has been the development of the vertical "floating zone" technique in which the metal is supported vertically from the ends without contact with a container, the narrow molten zone being maintained by virtue of the surface tension of the metal.

Heating in either case is usually done by induction and the refining is commonly performed in an inert atmosphere. Better control and narrower zones are claimed for electron beam heating, however, and this technique, which requires very high vacuum, gives the added advantage of purification through decomposition and vaporization of some impurities.

Zone refining has been reported on only two metals of the rare earth group. Gadolinium was purified by Kendall (10) using the conventional horizontal induction heating technique with a tantalum crucible in a purified argon atmosphere. Ten passes of a 1 3/4" wide zone at a rate of 1.5 inches per hour were employed. Evaluation of the product was solely by metallographic examination. This showed substantial concentration of impurities at the ingot ends with corresponding purification of the center of the ingot. Indications were that further refining by the technique would provide a pure metal, at least from the standpoint of microstructure.

Extensive effort has been expended by General Electric's Aircraft Nuclear Propulsion Department and General Engineering Laboratory, Battelle Memorial Institute, and Union Carbide's Oak Ridge National Laboratory on the zone refining of yttrium. This work has only recently been declassified, and no references are as yet available. The results in all cases, however, were in good agreement and may generally be represented by the experiments made at GE-ANPD. The "floating zone" technique was employed with induction heating, and a 3/8" diameter rod was processed in a continuously pumped vacuum. It was necessary to make three heating passes below the melting point in order to remove sufficient volatile impurities to prevent arcing to the heating coil. The deposit on the coil after this treatment was pyrophoric and was found to be mainly magnesium. That on the walls of the vessel was identified as yttrium oxyfluoride. Six zone passes were made at a rate of 2½" per hour.

Table II gives the results of analysis of the zone refined and original material. It is evident that no significant change in oxygen and nitrogen content was effected. There is some evidence of movement of a few of the metallic impurities, but this is not striking and may be questioned on the basis of reliability of the analytical techniques. Figure II shows the microstructure of the metal at three locations after the treatment. While structural changes are evident, none of the material can be classed as "clean". Hardness of the metal was measured at these same locations but showed no significant variation.

The general conclusion from these data and the similar results of other laboratories is that zone refining is not a practical purification process for yttrium. The indicated movement of metallic impurities, if real, is very slight, and excessive processing would be required for significant overall improvement.

Vacuum Processing for Refinement

The refining of metals by melting or vaporizing in vacuum is one of the oldest processes and has been the subject of much study (11, 12). The techniques employed range from simple melting in a crucible under vacuum to the more sophisticated methods of melting by electron bombardment and fractional distillation. The purification which results in any process is a function of the total pressure maintained and of the thermodynamic properties of the metal and the impurities themselves. Metallic impurities may, in theory, be separated by fractional distillation so long as their volatilities are different from that of the base metal. Non-metallic impurities, as noted earlier, are found in solution or more commonly as compounds. The former may be removed through volatilization, often at temperatures below the melting point. The latter may be removed by direct volatilization, by dissociation (either to an element which vaporizes or to a subcompound which is more volatile than the metal), or, where the compound is extremely stable and has low volatility, by distilling the metal away from the compound.

Melting Under Vacuum

The metallothermic reduction of the rare earth metals is customarily completed by a vacuum remelt which serves primarily to remove the retained reductant, usually calcium. Melting at pressures of 10^{-3} mm Hg is adequate for this purpose and reduction of Ca from about 1% to less than 150 ppm is obtained (2).

Similar purification is obtained in the case of yttrium by consumable electrode arc melting in vacuum. The pressures which can be maintained in this operation are not particularly low since it is difficult to stabilize an arc with low pressures, and since the considerable evolution of volatile material effectively increases the pressure in the vicinity of the molten material.

Table III gives comparative analyses for calcium and magnesium of yttrium metal arc melted in an argon atmosphere and in a nominal 10^{-3} mm Hg vacuum. The volatile metals have been effectively removed, but little significant change in other impurities was observed from this treatment.

Very significant improvements in the vacuum melting process have resulted from the development by Temescal Metallurgical Corporation of electron bombardment heating (13). Chief among these, from the standpoint of purification, is the ability to maintain pressures of the order of 10^{-4} mm Hg in the immediate vicinity of the melting metal. With such pressures, all volatilization reactions proceed more rapidly and to a greater degree of completion. Further, since the melting metal drips off the feed stock, a large amount of surface is exposed, and this also enhances purification reactions.

A theory has been proposed for the deoxidation of metals under high vacuum, in which the primary factor is the evolution of a volatile metal suboxide. The results observed on columbium, tungsten, titanium, and zirconium tend to substantiate this theory. With adequate thermodynamic data, the tendency for a metal to deoxidize should be predictable, but such data are generally lacking for the rare earth metals. It has been estimated that yttrium should deoxidize, however, and several attempts have been made by the Temescal Metallurgical Corporation to verify this. In no case has any significant deoxidation taken place, and in a few cases an increase in oxygen content was observed. Results of a typical melting are shown in Table IV. This failure to deoxidize may result from a breakdown of one of the basic assumptions in the case of yttrium, or from the tying up of oxygen by an impurity which is not itself deoxidizable.

Melting of other rare earth metals by this process has not been reported.

Purification by Distillation

As many of the rare earth metals have fairly high vapor pressures (14), it should be possible to purify them by distillation. In a few cases, e.g. samarium, europium, and ytterbium, where the usual metallothermic fluoride reduction was not feasible, the primary preparation was made at Ames by reduction of the metal oxide with lanthanum or misch metal in tantalum apparatus with simultaneous distillation of the volatile metal product. Dysprosium, erbium, thulium, and scandium have also been prepared in this way. The volatilities thus demonstrated suggest the direct distillation of the impure metals and this has also been reported by Ames (2), with a "considerable" reduction in oxygen content.

Trombe (15) describes the distillation of dysprosium using a tungsten condenser and a molybdenum crucible, the whole being enclosed in a pyrex evacuation chamber and heated by induction. A compact deposit "well freed from its impurities" was obtained, while a blackish residue of oxide containing silica and iron remained in the crucible. He indicates that lanthanum has been purified in the same manner, which is surprising in view of its low vapor pressure. No data are given on the analysis of these metals, but they are noted as being more malleable than the unrefined reduction products.

Petru (16) prepared scandium by metallothermic reduction and indicated further purification by distillation at 1500°-1600°C and 10^{-4} mm Hg. An identical preparation and purification of scandium was reported by Iya (17).

Daane (18) has prepared yttrium by direct distillation at approximately 1900°C in tantalum apparatus. Distillation proceeded readily, but the high temperature resulted in considerable contamination with tantalum at the condenser surface. Other impurities have not as yet been evaluated.

van Arkel - de Boer Deposition

The "iodide" or "hot wire" deposition process developed by van Arkel and de Boer in 1925 (19), has been applied with success to the purification of several of the "reactive" metals, the prime example being zirconium. The basic technique consists of heating the impure metal in a closed system with iodine. The metal reacts forming a volatile iodide which is decomposed on contacting a hot wire or surface, depositing purified metal and liberating the iodine for further reaction.

Loonam, in a recent review (20), defines two basic requirements for a metal which determine its applicability for refinement by this technique. The metal must: (1) be capable of existing as a solid or liquid at some temperature and pressure in equilibrium with a gas of high atomic ratio of iodine to metal, and (2) be capable of reacting readily with the gaseous products of the deposition reaction at the same pressure but a different temperature to yield a gaseous product of low atomic ratio of iodine to metal.

In addition, it is desirable that the metal have a high melting point and a low vapor pressure, but these aid in deposition as a solid and are not essential factors.

Ideally, the iodide of the metal should have a low heat of formation, but the process is feasible for many metals with high values if they form tetra or tri-iodides, since the large positive entropy changes on dissociation in such cases result in an overall decrease in free energy of dissociation with increasing temperature. On this basis, the metals least likely to be amenable to refining are those which form mono-iodides which remain monomeric and stable in the vapor state. Loonam predicts that the mono-iodides of the rare earth metals will be sufficiently stable to prevent dissociation, and this conclusion is confirmed by the excellent collection of thermodynamic data presented by Scaife and Wylie (21).

The generally high volatilities of the rare earth metals also work against their purification by deposition, since the high temperatures necessary for dissociation of the iodide will also result in vaporization of any metal deposited.

Deposition of lanthanum, the least volatile metal of the group, was attempted by Foster and coworkers (22) in a modified van Arkel - de Boer deposition in which the iodide was formed by reaction of iodine and aluminum on lanthanum oxide. Dissociation proved to be negligible at 1000°C and was only slightly improved at 1800°C. The metal was appreciably volatile at the latter temperature, and only small amounts of lanthanum were detected on the wire. The deposit was contaminated with aluminum from the iodide preparation.

Frazier (23) has attempted the deposition of yttrium from both bromide and iodide at bulb temperatures from 300° to 520°C and molybdenum filament temperature from 600°C to 1600°C. No deposit was obtained in any of the experiments.

Trombe (15) mentions that it has not been possible to deposit the rare earth metals by the van Arkel - de Boer technique, but gives no details of any trials.

Formation and decomposition of metal carbonyls has been utilized as a purification technique. Though similar in principle to the van Arkel - de Boer process, the reactions are usually carried out in separate chambers at different pressures. Data are entirely lacking on carbonyls of the rare earth metals, but one patent (24) claims purification of crude cerium by formation of the heptacarbonyl with CO under heat and pressure, followed by distillation and dissociation to metal and CO. This technique, if confirmed, would appear to be worthy of investigation for other metals of the group.

Solid State Electrolysis

The mobility of foreigh ions within a metal lattice subjected to a DC field has been recognized for many years (25), but little study of the phenomenon has been made. The effect is most pronounced for the non-metallic atoms which can presumably migrate interstitially in the metal, but transport of metal impurities has also been observed (26). The remarkable mobility of oxygen in zirconium was reported by de Boer and Fast (27), and this led to the consideration by General Electric's Aircraft Nuclear Propulsion Department of the technique as a purification method for yttrium (28). There is no indication in the literature of any prior use of the method for purifying metals, though Joffé (29) described the removal of metallic impurities from quartz in this way.

Yttrium bars of 3/8" and 3/4" diameters were electrolyzed in static argon by GE-ANPD at 2500°F and 2200°F respectively for 200 hour periods using the vacuum chamber of an arc melting furnace. Water cooled electrodes were attached to the bars, which were heated by the passage of direct currents of some 300 to 700 amperes. Figure III shows one of the bars at the completion of the electrolysis. A number of slight offsets or "kinks" are evident, and these were subsequently shown to be transverse grain boundaries between large single crystals of yttrium.

Results of the electrolysis were similar at the two temperatures employed, though greater overall transport of impurities was observed at the higher temperature, as would be expected for a diffusional process. Results of analysis at three locations on the 2200°F specimen are shown in Table V and are compared with the original bar. Marked movement of oxygen toward the anode was observed, with less pronounced movement of nitrogen in the same direction. A number of metallic impurities were also moved toward the anode.

Figure IV shows the microstructure at different locations in the bar. The inclusions of the original have been almost completely removed near the cathode. Figure V shows a diametric section through the anode end of the bar. The striking accumulation of inclusion material offers further evidence of the cleansing effect of the electrolysis. Diffraction patterns of this material indicate both Y_2O_3 and YOF. Hardness and electrical conductivity showed no significant variation over the bar except in this region of high inclusion concentration.

The pronounced transport of oxygen was surprising in view of its low solubility in yttrium. It must be concluded that the mobility of that which does dissolve is quite high, and that the process continues until the insoluble inclusion is all removed. Thus, it seems probable that similar purification of the other rare earth metals might be obtained by this technique, whatever their apparent solubilities for oxygen.

Miscellaneous Purification Techniques

A few purification techniques have been described which potentially are applicable to the rare earth metals, and which have been attempted with one or more metals of the group. One of these is the flotation of insoluble impurities from an amalgam either of the metal or its hydride. Schumacher (30) and Audrieth (31) reported direct amalgamation of the rare earth metals. As oxides, carbides, halides, etc. were insoluble in mercury, these impurities floated on the amalgam and could be separated by under-pouring or filtration.

Efficient use of this technique requires fine division of the metal, and this can be accomplished by forming the brittle hydride and grinding. Warf and coworkers (32, 33) have shown that mercury wets the hydrides of lanthanum, cerium, and ytterbium, and they mention that oxide and other impurity inclusions floated on the amalgam. Frazier (23) tried to wet YH_3 with mercury without success, however. Assuming the flotation to be successful, further processing by vacuum distillation would remove both mercury and hydrogen, leaving a purified metal powder.

The readiness with which the rare earth metals form relatively stable hydrides suggests purification by hydride precipitation from a low melting alloy through introduction of hydrogen. The hydride would then be separated from the melt by filtering or decantation and further treated by vacuum distillation to remove hydrogen and mercury and produce metal powder. This technique has been employed by Woerner and Chiotti for thorium (34). Frazier bubbled hydrogen through a low melting alloy of yttrium, aluminum and magnesium (23). The results were inconclusive due to excessive frothing of the melt, but indicated that extreme difficulty would be encountered with this system in the separation step, and no further work was done.

An electrorefining technique developed by the Bureau of Mines Electrometallurgical Experiment Station has been used with success for purification of a number of reactive metals (35). The impure metal is made the anode in a molten salt bath, and purified metal is deposited as dendritic crystals on the cathode. Experiments are currently in progress at the Station on the refining of yttrium in this manner, and early results show encouraging reduction of oxygen content of the metal (36). It seems reasonable to assume that this process might be used to advantage with other metals of the rare earth group.

Conclusions

Behavioral differences within the rare earth metal group are sufficiently pronounced to make generalizations in regard to purification of questionable value. In the few cases where the same technique has been applied to two members of the group, the results as often as not have been contradictory. Nevertheless, it would appear that those techniques which are generally most promising for purification of the rare earth metals are distillation and solid state electrolysis. Zone refining may be of value for some impurities, but the van Arkel - de Boer deposition technique appears to hold little promise. None of the techniques with the possible exception of amalgamation is particularly attractive as a production process, and it must be concluded that refinement of the reduction itself is the optimum method for obtaining a pure metal in quantity. A similar conclusion was reached by Richardson (37) in a recent theoretical survey of metal refining techniques.

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TABLE I

Typical Analysis of Yttrium Metal Ingot*

Element	Wt. %
Carbon	0.011
Nitrogen	.019
Oxygen	.275
Hydrogen	.009
Fluorine	.075
Iron	.062
Nickel	.030
Calcium	.0005
Magnesium	.002
Silicon	.002
Copper	.004
Aluminum	.006
Chromium	.013
Boron	<.001
Zirconium	.580
Total Rare Earths	98.87%

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* Produced by Ca-Mg reduction of YF_3 in zirconium vessel, followed by vacuum "demagging" and consumable electrode arc melting in argon with remelt in vacuum.

TABLE II

Results of Zone Refining of Yttrium by GE-ANPD

Impurity	Content in Metal Before Processing, ppm.	Content in Metal After Processing, ppm.		
		Near Beginning of Zone	Center	Near End of Zone
Oxygen	5800	7600	6170	5310
Nitrogen	270	280	250	250
Iron	500	500	300	100
Chromium	10	10	< 1	< 1
Copper	10	2	0.5	5
Nickel	50	50	50	50
Titanium	100	100	100	100
Zirconium	4700	4300	3100	2750

TABLE III

Comparative Removal of Calcium and Magnesium from Yttrium Metal by Consumable Arc Melting in Argon and Vacuum

Melting Procedure	Analyzed Impurity Content, ppm					
	Calcium			Magnesium		
	Max.	Min.	Avg.	Max.	Min.	Avg.
Two melts in Argon*	800	50	350	800	100	300
First melt in Argon, second melt in 10^{-3} mm Hg vacuum**	6	1	5	30	6	23

* Average of results on 17 ingots.

** Average of results on 25 ingots.

TABLE IV

Results of Electron Bombardment Melting of Yttrium Metal by Temescal Metallurgical Corporation*

Impurity	Content in Original Metal, ppm	Content in Processed Metal Ingot, ppm		
		Top	Center	Bottom
Oxygen	3250	3500	3115	3470
Nitrogen	200	200	210	310
Zirconium	5000	6000	5000	4000
Chromium	150	300	200	100
Iron	1000	620	450	190
Nickel	2000	2000	2000	2000
Copper	35	300	100	100

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* Analyses made by General Electric - Aircraft Nuclear Propulsion Department.

TABLE V

Results of Solid State Electrolysis of Yttrium Bar

2200°F for 200 Hours

Impurity*	Content in Metal Before Processing, ppm.	Content in Metal After Processing, ppm.		
		Near Cathode	Center	Near Anode
Oxygen	3330	665	3100	10900
Nitrogen	510	366	570	700
Silicon	55	100	10	140
Iron	150	60	50	600
Manganese	9	< 1	< 1	6
Magnesium	5	10	< 5	6
Zirconium	9000	9000	9000	7000
Nickel	250	50	100	1000
Chromium	80	20	20	30
Boron	7	3	6	15
Titanium	9	< 3	< 3	30
Cobalt	1	< 1	< 1	6

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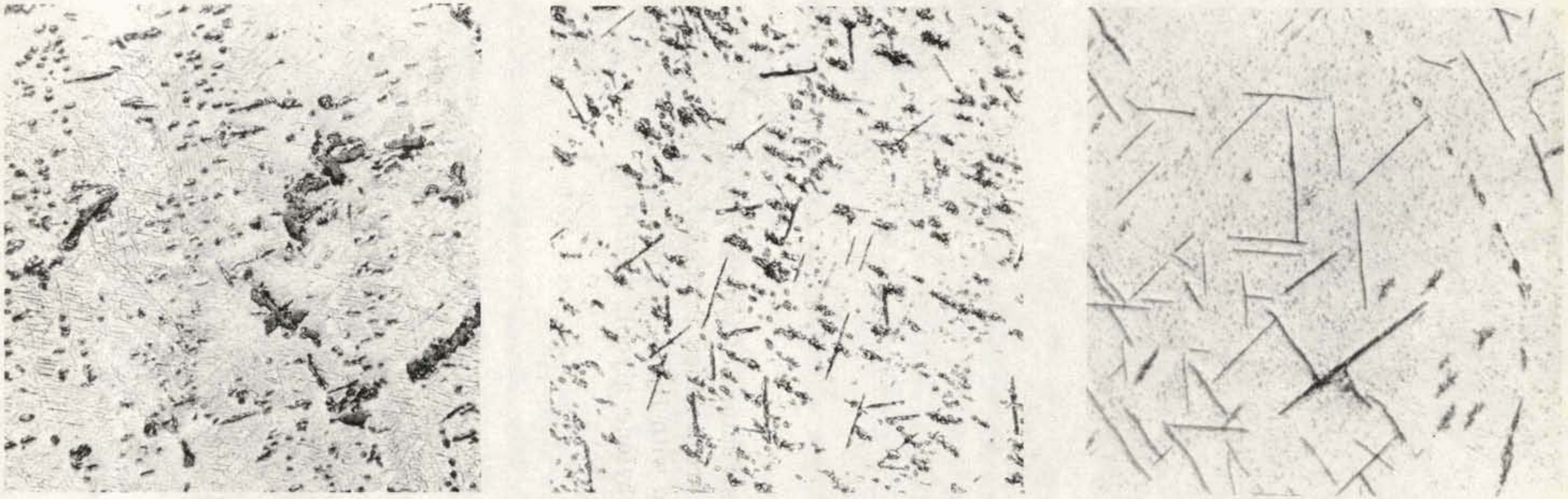
* Other impurities showed no change as a result of the electrolysis.

Fig. I - Microstructure of typical yttrium metal from Ames Process. Etched with H_3PO_4 in glycerin and ethylene glycol. (X250)



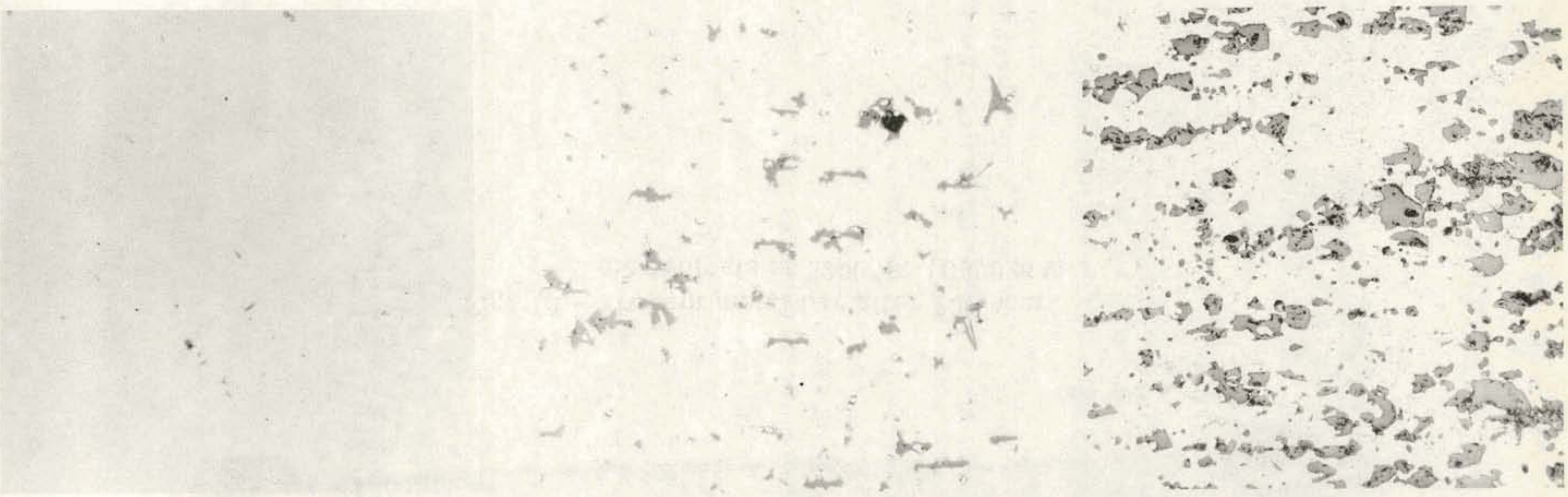
Near start of zone

Center

Near end of zone

Fig. II - Microstructure of zone - refined yttrium metal at three locations. Etched with H_3PO_4, HNO_3, and CH_3COOH. (X500)

Fig. III - Yttrium metal bar after 200 hour electrolysis at 2200° F. (U37026A)



Near Cathode

Center

Near Anode

Fig. IV - Microstructure of electrolyzed yttrium bar at three locations. Unetched. (X250)

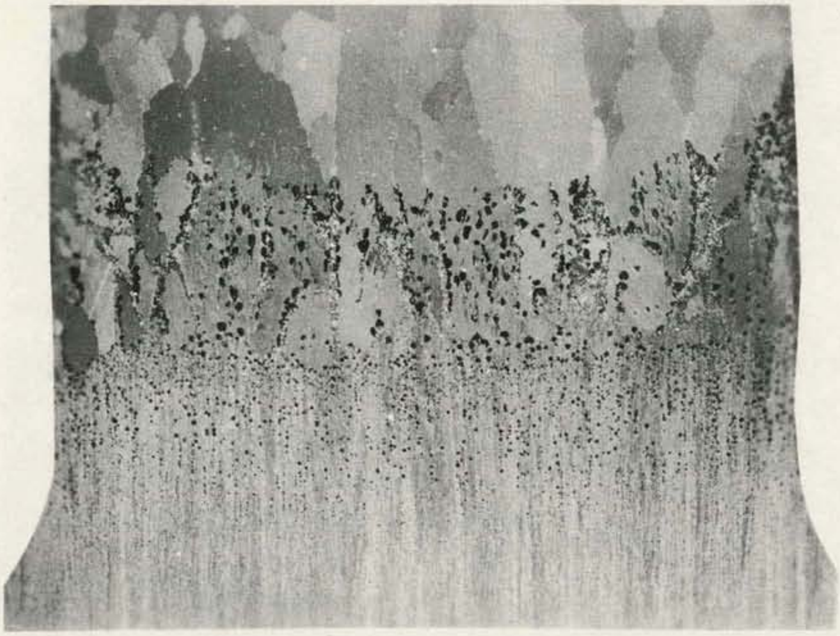


Fig. V - Diametric section of anode end
of electrolyzed yttrium bar.
Unetched. (X5)