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THE EFFECT OF RARE EARTH ADDITIONS ON LOW ALLOY STEEL

BY

SUKH DEV SEHGAL

A

THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

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Degree of

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Approved by



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INTRODUCTION

Rare earth metals have for some years been used as an inoculant in nonferrous foundry practice and lately in the production of nodular iron. It has been reported that addition of rare earth metal to certain grades of stainless steel so improve the hot malleability that reduction of large size ingots in the blooming mill is now possible. It has been reported that considerable improvement in soundness and mechanical properties of steel castings and wrought products have been attained as a result of small additions of rare earth metals and oxides. In practically all investigations, it has been reported that most of the properties such as workability, strength, impact properties, and oxidation resistance, improve by the addition of about two pounds per ton of rare earths. Some of the authors consider rare earth metal additions as a penicillin for steel castings in solving production difficulties. There have been some contradictions as to the degree of improvement affected by rare earth additions, but none of the useful properties of steel have been found to be impaired. The American Metallurgical Products Company of Pittsburgh, Pennsylvania has even recommended a new system of steels being developed with rare earth additions.

The present investigation was undertaken to extend the information on improvement in properties and to find some causes of the drastic effects of rare earths. The work has been confined to low alloy steel composition, which is best suited for the production of steel castings. The various

phases of the study included grain size determinations, desulfurization effects, as also the examination of inclusions, all of which affect properties of steel considerably. The properties of rare earth metals have been studied and an effort made to analyze the thermodynamic possibilities of their action. The use of fluorides of rare earths was included to see if they have the same effect as oxides and metals.

The investigation will be useful to show the effects more clearly on the steel quality, by the addition of rare earth elements, and may help in deciding upon the application of these elements for certain specific steels.

REVIEW OF LITERATURE

Some of the earliest information available on the use of rare earths was given by W. Hessenbruch, a German professor. He reported in 1938 that these elements imparted remarkable increase in life of resistance wire in a variety of alloys. Their application to the nonferrous industry dates back to the 1920's when, in Germany, they were employed to get high strength aluminum alloys for the aircraft industry, and since then extensive investigations have been made in various nonferrous metals and alloys.

Cerium has been used for some years in the production of nodular cast iron and a number of papers have been published on the subject since the early 1940's. The use of cerium was tried in 1947 by E. C. Piggott¹. He observed slight improvement in tensile and bending properties of steel. The action of cerium as a scavenger element was also realized.

For some years no work was done and a systematic investigation was started again in 1951. C. B. Post, D. G. Schoffstall and O. H. Beaver² reported the effect of adding misch metal to austenitic chrome nickel steels containing nitrogen. They found improvement in hot ductility and hot workability and noted that nickel content determines the amount of cerium and lanthanum needed. The same year the above authors were granted United States Patent Number

¹All references are in bibliography.

2,553,330.

Another team of investigators during 1951 report the effect of cerium on impact strength of steel. C. D. Berry and A. A. Dorvell³ relate the improvement of impact to the amount, shape and distribution of non-metallic inclusions.

The period after 1951 has been marked with a great deal of activity in this field. An extensive paper in 1952 by G. A. Lilliequist and G. C. Michelson⁴ describes the effect of rare earth additions on cast steels.

LanCerAmp was used in these investigations and heats were made in high frequency induction furnace. They established the following results: LanCerAmp did not eliminate temper brittleness, but the reduction in area and charpy values of treated steels were much higher than untreated steels in the temper brittleness zone of temperature. Although LanCerAmp changed the shape of inclusions from a crystalline shape to a rounded shape, the great improvement on charpy tests showed that some other factor was also at work. This other factor could not be determined. The fluidity of the melt was improved; the possibility of cracking during hot working and forging was decreased; LanCerAmp seemed to aid in sulphur removal; the solidification temperature of steel was lowered; the hardenability was improved; and no difficulty in welding was noted.

During the same year W. E. Knapp and W. T. Balkcom⁵ made similar investigations with LanCerAmp additions, containing more lanthanum than does misch metal. Desulphuri-

zation and other benefits for steels are cited, i.e. better low temperature impact properties, increased reduction in area, higher temperatures at which grain coarsening occurs, and better hot and cold workability. LanCerAmp lowers the impact transition temperature by 100° F. A maximum in hardenability falls off with increased additions.

C. B. Post and O. H. Beaver⁶ made further investigations using rare earth oxides, fluorides and misch metal. It was found by them that the mechanism of misch metal additions and the rare earth oxide mixtures are different. The rare earth oxide mixtures do not necessarily improve the hot workability, but are better and cheaper than misch metal additions. The recovery of cerium and lanthanum from the commercial rare earth oxide mixture is small, if not absent. Cerium fluoride additions to the ladle in the molds have been found to improve the ingot surface. While a cleaner steel is obtained by the addition of the rare earth oxides, no significant improvement was noticed except with a steel having an improperly made misch metal addition.

C. Wells, J. V. Russell and S. W. Poole⁷ studied the effect of lanthanons as oxides and metal on steel ingots. They found that lanthanum oxide additions decreased the hardenability effect of boron on steel but did not significantly affect other steels.

Desulphurization of steel by rare earths has been studied by M. G. Snellman.⁸ He found the desulphurization effect to be strong in the first few minutes of the addition. Sul-

phur reversion result from the oxidation of the rare earth sulphides floating on the melt and subsequent diffusion of the free sulphur back through the melt. Behavior under air and reducing gas was similar. A report on desulphurization with rare earths appears in Steel.⁹ Induction melting with fifty pounds heat was practiced in the investigation and, in addition to M. G. Snellman's⁸ report, it contains the following important features: (a) Under oxidising or air atmosphere, sulphur reversion occurs until practically the original sulphur content is restored. Under neutral or natural gas atmosphere, limiting sulphur, content after reversion was lower than the original content. (b) Rare earth misch metals (La-metal and 80% La containing LanCerAmp) produced practically equivalent desulphurization under test conditions. (c) X-ray diffraction analysis indicate a CeO_4 type crystalline component as one of the constituent phases in slag scum samples obtained after sulphur reversion takes place.

A. J. Bager,⁹ R. H. Henke¹⁰ and Norman F. Tisdale⁹ gave information seperately on various effects of rare earth additions. R. H. Henke reported that the rare earth oxide compounds did not produce segregated areas that were common to the metal additions. While positive evidence of production of fine grain steel was not found, he recognized enhanced hot workability. N. F. Tisdale told that oxide compounds do not remove inclusions but scatter them so they do not cause harm.

The effect on the surface quality of low carbon steel by rare earth additions was studied by J. V. Russel.¹¹ He reported desulphurization caused by lanthanon addition and also that the efficiency of removal of sulphur increases with increased sulphur content. Improved surface quality was obtained at low manganese-sulphur ratios. Addition to aluminum-killed steel in amounts exceeding two pounds per ton resulted in a hot short condition, causing hot tears in rolling. Lanthanon oxides, on the other hand, reduce the effect of aluminum.

O. H. Beaver¹² again reports the effect of rare earth additions on steel. He says that though not a panacea to all steel problems, the lanthanons do have a place in the production of wrought alloys. He reports that scaling resistance up to 1900° F was improved. When tramp elements cause difficulty in working, lanthanons especially improve the hot workability. The mechanism depends on grain nucleation and removal of oxygen and sulphur.

H. Swartzbart and J. P. Sheehan¹³ studied the effect of lanthanon additions on transverse impact properties. They observed that lanthanon does not affect the maximum energy in a clearcut way, as does sulphur. Three split heats were used in evaluating the effect of lanthanons in the presence of high and low sulphur, high and low hydrogen, and high and low nitrogen contents. Impact strengths were doubled in the low sulphur steel and the effect of nitrogen was countered in the high nitrogen steel. There was little effect on the

High and low hydrogen steels.

G. Oesterberg¹⁴ tried to dephosphorize cast iron and steels by adding calcium and obtained partial dephosphorization. Misch metal, when added by an amount of 6.6% to cast iron, gave 19% phosphorus removal. The method is uneconomical for general use at the present time.

J. W. Sprotnak¹⁵ gives information about the fields of application of rare earth salts. These are used for projectors and searchlights, flints for cigarette lighters, additives in steel making, filter cloth for bauxite, lens polishing and special glass. Europium, gadolinium and samarium have neutron absorption properties of interest to physicists.

W. J. Jackson¹⁶ has reported similar results with oxide, fluoride and misch metal additions. He reports that oxides and fluorides are more affective for improving the hot workability than the metal. The best addition of misch metal for ductility, fluidity and impact properties is from two to four pounds per ton of steel.

V. M. Tageev and Yu D. Smirnov¹⁷ have reported the prevention of whisker during crystallization. Whisker formation, i.e. accumulations of sulphur and other impurities due to differences of solubility in the solid and liquid phases of solidifying steel, can be affectively avoided by treating liquid steel with a mixture of rare earth elements in amounts of 0.01 to 0.20%. In the presence of these elements, the separation of sulphide phase occurs at earlier stages of

crystallization and is therefore more uniformly distributed.

In another publication in 1957, W. E. Knapp¹⁸ and others have summed up the effects of rare earth additions and they report as follows:

1. Prevention of hot shortness in high alloy austenitic stainless steels.
2. Tramp elements controlled.
3. Promotion of nodular formation in the manufacture of ductile iron.
4. Impact resistance improved in cast steels and low alloy wrought steels.
5. Control of oxygen, hydrogen and sulphur in solidifying metal.
6. Scale suppression in forging.
7. Refinement of ingot grain structure.

As an example of improvement in impact and wear resistance properties of rare earth treated steel, Wearpact steel (produced by American steel foundries) is coated. The present cost of rare earth metals is considered to be a handicap in their application for deoxidation of steel.

W. G. Wilson¹⁹ has reported a summary of recent Russian papers published concerning the elimination of inverted V segregation by the addition of one to four pounds of misch metal. In untreated steel ingots the sulphur seemed to stay longer in solution, giving it more chance to segregate into dendritic boundaries. They report that removal of sulphur from solution seemed to increase the melting point of other inclusions. Therefore, elimination of sulphur from the system has more of a compounding effect rather than its being a straight-forward effect. The effect of rare earths on the melting points of sulphides has been studied and the work shows that rare earths, when mixed with FeS raised melting

points anywhere from 30° to 270° F.

The Russians¹⁷ found that rare earth treatment makes the ingot skin more homogeneous but sulphide precipitations to be larger behind the skin. Their work shows that addition of as small as 0.25% Ce, increases fluidity by 30%, but if the iron is allowed to set for as little as three minutes, the fluidity begins to fall rapidly. Their explanation for these phenomena has to do with what they call emulsion of cerium oxide which forms through oxidation of the relatively large amount of misch metal added. This high melting point oxide, they feel, lowers the fluidity.

Study of nonmetallic inclusions has been made by Perkins²⁰ by deoxidation of steels with different deoxidizers, including misch metal. He investigated the tensile ductility of ingot steel at 2400° F after deoxidation with Si, Al, Ti and rare earths and observed significant improvement in this property. Oxidation practice was found to affect residual oxygen content and changed the nonmetallic inclusions to different types, characteristic of the specific element added.

RARE EARTH METALS AND COMPOUNDS

The rare earths are the elements of atomic numbers 57 to 71, beginning with lanthanum and cerium and ending with lutetium. The first clean separation or discovery was of cerium by Berzelius in Sweden in 1803. Last was that of promethium by a team of American researchers in 1947. Their chemical properties are so similar, that separation of high purity elements were almost unknown until during the 1940's. For many years the rare earths were the subject of purely academic interest to only a few chemists. Then almost overnight the atomic bomb created a great interest in the properties of rare earth elements, because they form such a high percentage of the ashes of nuclear fission.

Since the term "rare earths" implies some scarcity of these elements, it is of interest to consider the relative abundance of each one. Table No. 1 gives the average weight percentage of each element in the rare earth content of all known rare earth ores, and also the percentage of each in the earth's lithosphere. The ^{even} members are more abundant than their odd atomic-numbered neighbors. Cerium makes up nearly one-third of the whole group and it has found more commercial applications than the other members.

The principal source of rare earths is the mineral monazite found as an alluvial sand in Brazil, Travancore in India, and Idaho. This is essentially a complex phosphate, containing often as much as 70% cerium with other rare earths and 6 to 9% of thorium. Other important mineral sources are

TABLE NO. 1
RELATIVE ABUNDANCE OF RARE EARTHS

<u>Rare Earth</u>	<u>Percentage of Group</u>	<u>Per cent of Earths crust</u>
La 57	7	0.00035
Ce 58	31	0.00155
Pr 59	5	0.00025
Nd 60	18	0.0009
Pm 61	?	?
Sm 62	7	0.00035
Eu 63	0.2	0.00001
Gd 64	7	0.00035
Tb 65	1	0.00005
Dy 66	7	0.00035
Ho 67	1	0.00005
Er 68	6	0.00030
Tm 69	1	0.00005
Rb 70	7	0.00035
Lu 71	1.5	0.00007

gadolinite, samarskite, xenotime, Yttrocerite, cerite and allanite, The procedure for separation of rare earths from each other most utilized has been fractional crystallization; only recently has this tedious method been improved upon by the introduction of separations depending upon the oxidation or reduction of some of the elements to other than the tri-positive state. Solvent extraction and ion exchange are other processes extensively applied to rare earth extractions. In discussing separations, the rare earths are divided into two groups - the cerium group and the ytterium group. The cerium group includes the elements La, Ce, Pr, Nd, Sm, and Eu; the ytterium group includes Y, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu. Table No. 2 gives the properties of the rare earth metals. Thorium, which is always present in monazite, is separated from rare earths by differential dissolution of the rare earth oxalates, leaving thorium oxalate undissolved. Many other physical properties are given by Yost,²¹ Russell and Garner.

Properties

All rare earth metals possess an unusually strong chemical affinity for the nonmetallic elements, normally associated with steel: sulphur, oxygen, hydrogen, nitrogen and carbon. In addition, they alloy readily with all common metals, in many cases forming significantly refractory intermetallic compounds with the parent metal. With many possibilities for reaction with the various constituents in steel, it is not surprising that unequivocal explanation of the behavior of rare earths has not appeared. Table No. 3 and 4 show the

TABLE NO. 2
 PROPERTIES OF RARE EARTH METALS

<u>Element</u>	<u>Symbol</u>	<u>Atomic No.</u>	<u>Density</u>	<u>Melting point</u>	<u>Boiling point</u>	<u>Sp. heat</u>
Lanthanum	La	57	L - 6.194 B - 6.18	1580	3275	0.045
Cerium	Ce	58	L - 6.78 B - 6.8	1420-1470	2550	0.042
Praseodymium	Pr	59	L - 6.778 B - 6.805	1725-1760	-	0.048
Neodymium	Nd	60	L - 7.004	1475-1650	-	0.045
Promethium	Pm	61	-	-	-	-
Samarium	Sm	62	6.94	2460	-	-
Europium	Eu	63	5.244	2000-2200	-	-
Gadolinium	Gd	64	7.948	-	-	-
Terbium	Tb	65	8.332	-	-	-
Dysprosium	Dy	66	8.562	-	-	-
Holmium	Ho	67	8.764	-	-	-

(continued)

<u>Element</u>	<u>Symbol</u>	<u>Atomic No.</u>	<u>Density</u>	<u>Melting point</u>	<u>Boiling point</u>	<u>Sp. heat</u>
Erbium	Er	68	9.164	2280		
Thulium	Tm	69	9.346	-		
Ytterbium	Yb	70	7.01	3275		
Lutetium	Lu	71	9.74	-		

TABLE NO. 3
 PROPERTIES OF RARE EARTH OXIDES

Compound	Density	Heat of Formation Formula wt. cal/gm mole at 1500° C	Melting Point °C	Boiling Point °C
La ₂ O ₃	6.51	-339,000	4200	7800
Ce ₂ O ₃	6.9-7.0	-337,000	3075	
Nd ₂ O ₃	7.24	-	-	
Pr ₂ O ₃	6.88	-336,000	-	
Fe ₃ O ₄	-	-153,000	-	
FeO	-	-38,000	1375	
MnO	-	-67,000	1800	
Al ₂ O ₃	-	-291,000	1280	
MgO	-	-102,000	-	

TABLE NO. 4
 PROPERTIES OF SULPHIDES

<u>Compound</u>	<u>Heat of Formation per Formula wt (cal/gm)</u>	<u>Melting Point OF</u>	<u>Color</u>
La_2S_3	-317,330	3800-3900	Redish yellow crystals
Ce_2S_3	-	3500	Red crystals
Nd_2S_3	-262,610	4000	Green powder
Sm_2S_3	-	3450	Yellow pink
Yb_2S_3	-	3450	
Li_2S_3	-115,400		
Ca S	-113,500		
Mg S	-82,200		
Na_2S	-89,200	1700	
Mn S	-60,000	2768	
Fe S	-23,000	2180	

properties of oxides and sulphides of rare earths and some other elements. The normal reaction products of the rare earth metals with sulphur are sesquisulphides (Re_2S_3). From the heat of formation of these sulphides, it is obvious that rare earth elements are strong desulphurizers. Also the rare earth metal sulphides melt relatively at higher temperatures, well above those of molten steel. Being refractory, these complex sulphides occur as spheroidal, well dispersed particles and do not develop elongated typical sulphide inclusions during rolling, as do the sulphide inclusions in the untreated steel.

Rare earth metals have a strong affinity for hydrogen and the detrimental effect of extremely small percentages of that element in steel are well established facts. Reaction between rare earth and hydrogen are opposite that between iron and hydrogen. The former occlude the gas exothermically (as also do Tr, Zr, Cb, Ta, U, Pd); iron is an endothermic occluder. Regardless of temperature the rare earth metals are capable of dissolving tremendously greater quantities of hydrogen than can iron. Table No. 5 shows the solubility of hydrogen at one atmosphere, for different metals. At 2000° F, the solubility of LanCerAmp for hydrogen is 2000 times greater than that of iron. At 1475° F, solubility is 6000 times and at 750° F, it is 60,000 times greater than that of iron. Although the melting points of Ce, La, and Pr are 1450, 1435 and 1750° F, the melting points of the rare earth-hydrogen alloys are well above their melting points.

TABLE NO. 5
 SOLUBILITY OF HYDROGEN AT 1 ATM (CCS OF H₂/gm OF METAL)

<u>Temperature</u>	<u>Fe</u>	<u>La</u>	<u>Ce</u>	<u>Pr</u>
2912	-	-	-	-
2795	.267	-	-	-
2795	.34 (solid)	-	-	-
2200	.079	-	55	-
2000	0.054	113	113	125
1700	.045	132	130	138
1650	0.03	146	144	147
750	.003	190	181	179
86	-	230	218	210

Rare earth metals also have a strong affinity for nitrogen and carbon. Yellow crystals of rare earth carbides can be obtained by the reduction of oxide by carbon in electric furnace. - These carbides react with hot and cold water evolving a complex mixture of gases, of which acetylene is the principal constituent. The rare earth elements react readily with oxygen, forming very stable refractory oxides. Rare earth elements have a much greater atomic volume than most other common metals, and Table No. 6 shows the relative atomic volumes.

Important Applications

The pyrophoric properties of cerium alloys have made them of some use in the removal of oxygen from cast irons, in tracer bullets and luminescent shells which show their path by the light evolved on ignition of the alloy and in cigarette lighters. Cerium dioxide has been used as a fine abrasive for polishing glass and soft metals. In the early 1920's most important use of rare earths was in the gas mantle industry. In glass, ceramic and enamel industries, uses were found for pure and group compounds as opacifiers and colorizers. Rare earth oxides and fluorides are used in cores in arc carbons and provide a white light simulating day light, used widely in the motion picture industry.

The most common form in which these metals are furnished for metallurgical use, is the alloy called "misch metal". The approximate composition is Ce-50 to 55%, Nd-15 to 17%, and other rare earths 8 to 10%. The term rare earth oxides refers

TABLE NO. 6
ATOMIC VOLUMES

<u>Element</u>	<u>Volume</u> <u>Cm³/gm</u> <u>atom</u>
Fe	7.10
Mn	7.4
Mo	9.4
W	9.55
Ce and Nd	20.5
La	22.6
S	15.5

to a mixture of oxides in a similar proportion. The metallurgical industry normally uses a mixture of rare earth oxides or metals due to economic reasons. Except for the use of cerium in cast iron, very little work has been done on the application of individual rare earth elements.

In Germany during the early 1920's it was found that misch metal addition to Al aircraft piston alloys improved creep and high temperature oxidation resistance, refined the grain and gave better mechanical properties at room temperature. Similar additions in Mg-base alloys gave comparable results. In United States the application of rare earths started for Mg and Al base alloys in the early 1940's.

The steel industry has for some time been using LanCerAmp as a rare earth addition. Two grades of LanCerAmp are usually available. The LanCerAmp No. 23 consists of 25% misch metal, 10% zirconium, with the balance being low carbon iron. It is the favoured combination for commercial use and has been found to be most consistent. The LanCerAmp No. 2 is substantially straight misch metal with 4% iron to lower the melting point from 1550 to 1250° F.

EXPERIMENTAL PROCEDURES AND RESULTS

Making of Steel Heats

In the present investigation low alloy steel heats were prepared in the induction furnace. Since in induction heating, the heat is entirely generated in the charge itself, melting is rapid and there is only a slight oxidation of scrap. The Armco iron punchings, which was the starting material for the heats were packed in the silica or magnesite crucible and the current was switched on. After about 30 minutes a pool of molten metal was formed at the bottom of the crucible and the charge sank. Additional punchings were then added.

Normally no attempt is made in induction furnace to melt under a slag cover, since it is difficult to keep a slag blanket, because of the convexity of bath resulting from the stirring action. Oxidation from atmosphere is so slight that a slag is not considered necessary for protection.

During melting the furnace top was covered with a refractory brick to conserve heat. Small 4 lbs. crucibles were used for the purpose. The crucible after being placed in the coil was held in position firmly by ramming fine sand round it and sealing the top by a mixture of sand and bentonite. The composition of Armco iron is as follows: C - 0.015%, Mn - 0.028%, P - 0.005%, S - 0.025%, Si - 0.003%, and remainder iron. (The specifications indicate maximum values of 0.010% P, 0.030% S, 0.15% C_{max}, 0.10% total of C, Mn, P, S and Si).

Armco iron was first shot blasted for 30 minutes to remove the rust from the surface. A total of 17 heats have been produced. The first three heats were made to determine approximately the amount of Fe-Si, electrolytic Mn and Al required to properly deoxidise the steel. The melting operation was found to be satisfactory and these heats were cast in square sand molds. Metallographic samples prepared from cast and annealed ingots were examined for porosity, inclusions and to assess the carbon content of these heats. The next two heats were prepared with high Cr, Mn, and C contents and deoxidation was done by Fe-Si followed by Mn and .1% Al. The heats produced were clean. The details of first five heats are given in appendix. . The pouring temperature was 3000° F. The charge calculations were then made for the low alloy steel. The composition that was aimed at for the next heats was as follows: C - .1 to 0.15%, Mn - 0.8 to 1.0%, S and P - low, Si - 0.7%, Cr - 0.5%.

Except for the rare earth elements the amount of materials used for deoxidation and alloying were calculated for 1700 gms of Armco iron melted, and these were as follows:

High grade Fe-Si (70% Si) used each heat	22 grams
Electrolytic Mn used each heat	22 grams
Low carbon Fe-Cr (65-70% Cr) (assuming 80% recovery)	14 grams
Graphite Carbon used each heat	2 grams
Al foil for final deoxidation and wrapping the rare earths	2 grams

Order of addition of deoxidisers and alloying elements

was as given. Fe-Si was added first, followed by Mn addition. The temperature at this stage was 3000° F. The slag formed at the surface was removed and then Fe-Cr and Carbon were added. The rare earth oxides, fluorides, or the LanCerAmp were now introduced in the crucible and the power switched off. The heat was tapped quickly after this addition. Table No. 7 gives the description of rare earth additions in different heats.

With the fresh crucible the time taken for the heat was 1¼ hour but the latter heats were tapped in 45 minutes time. An optical pyrometer was used for temperature measurements, and all heats were tapped at 3000° F. Except for No. 6 and 7 heats which were cast into dry sand mold for getting square ingots, all other were cast in cylindrical graphite molds. This gave longer length of ingot for investigation as the relative length of pipe was less. Metallographic samples prepared showed no carbon increase near the surface, presumably due to quick cooling and solidification. An instruction sheet about LanCerAmp induction practice was obtained from the American Products Company and effort was made to follow it after necessary modification. In between deoxidation and rare earth addition, slag was removed from surface. The metal was tapped after about two minutes of rare earth addition. The power rating of the furnace was maintained at nearly 10 KWH.

The composition of the different heats has been found spectrographically and is given in Table No. 8. This has been provided by the Courtesy of Ladish Company, Cudahy, Wis. Figures

TABLE NO. 7
RARE EARTH ADDITIONS IN THE STEEL HEATS

<u>Heat No.</u>	<u>Grams of Rare Earth Added</u>	<u>Form</u>	<u>Crucible Used</u>
6	No Addition		Magnesite
7	4½ gms.	Sm and Gd oxides	Magnesite
8	9 gms.	Sm and Gd oxides	Silica
9	13½ gms.	Sm and Gd oxides	Silica
10	18 gms.	Sm and Gd oxides	Silica
11	2.9 gms.	Rare Earth fluorides	Magnesite
12	8.9 gms.	Rare Earth mfluorides	Magnesite
13	23.2 gms.	Rare Earth fluorides	Magnesite
14	34.8 gms.	Rare Earth fluorides	Magnesite
15	7.5 gms.	LanCerAmp No. 23	Magnesite
16	22.5 gms.	LanCerAmp No. 23	Magnesite
17	45 gms.	LanCerAmp No. 23	Magnesite

TABLE NO. 8
CHEMICAL ANALYSIS OF STEEL HEATS PRODUCED

<u>Element</u>	<u>Heat No.</u>						
	<u>6</u>	<u>8</u>	<u>9</u>	<u>12</u>	<u>15</u>	<u>16</u>	<u>17</u>
C	0.13	0.10	0.12	0.10	0.12	0.12	0.11
Mn	0.89	0.84	0.86	0.96	0.99	0.95	1.02
P	0.008	0.008	0.007	0.008	0.007	0.008	0.007
S	0.019	0.014	0.012	0.017	0.015	0.016	0.014
Si	0.70	0.61	0.16	0.10	0.60	0.55	0.72
Ni	0.02	0.02	0.02	nil	nil	nil	nil
Cr	0.48	0.48	0.48	0.51	0.52	0.48	0.46
Mo	nil	nil	nil	0.01	0.01	nil	0.02
Co	0.01	0.01	0.01	0.04	0.06	0.95	0.05
V	0.011	0.011	0.011	nil	nil	0.01	0.02
As	0.013	0.01	0.011	-	-	-	-
Sn	nil	nil	nil	-	-	-	-
Cb	nil	nil	nil	-	-	-	-
Ta	nil	nil	nil	-	-	-	-
Zr	nil	nil	nil	Trace	Present	Present	Present
Ti	nil	nil	nil	-	-	-	-
Sm	Nil*	Nil*	Nil*	-	-	-	-
Gd	nil	nil	nil	-	-	-	-
Al	-	-	-	Trace	Present	Present	Present

* Sm and Gd were of quantities not detectable by routine spectrographic procedures.

Aluminum and Zirconium present in increasing increments in samples 12 thru 17.

No. 1a and 1b show the structure of ingot of heats No. 6 and 7, as cast.

Figure No. 2 shows the high frequency induction furnace used for making steel heats.

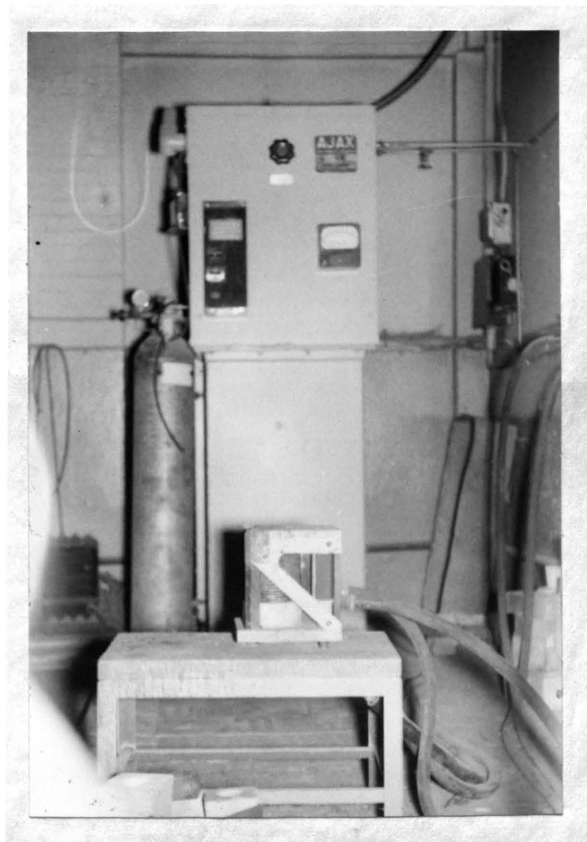


FIGURE 2
HIGH FREQUENCY INDUCTION FURNACE



FIGURE 1A
CAST STRUCTURE, NO. 6 HEAT
250x
3% NITAL ETCH 8 SECONDS

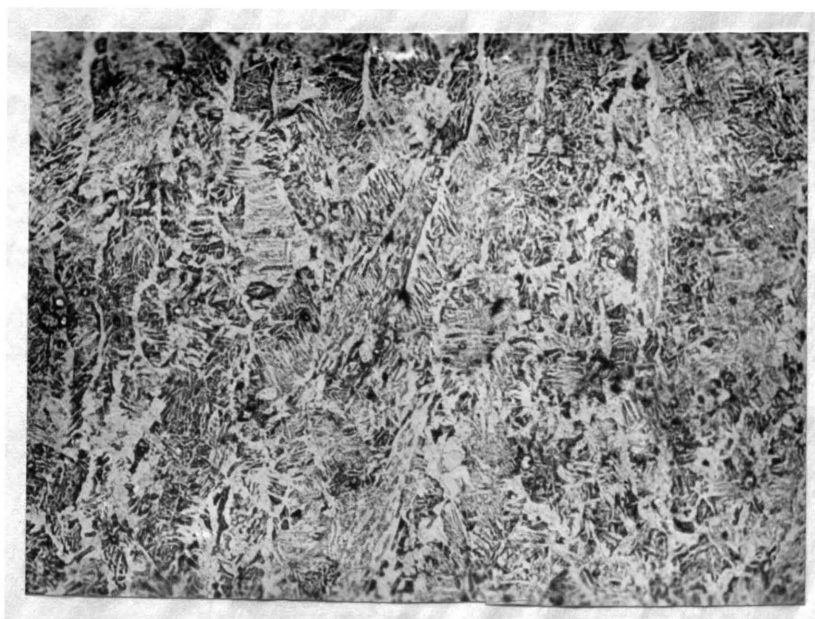


FIGURE 1B
CAST STRUCTURE, NO. 7 HEAT
100x
3% NITAL ETCH

Hardness Measurements

Due to the limitations of getting the tensile test pieces made at the Missouri School of Mines, only brinell hardness tests could be made. Originally it was thought to correlate these hardness values to tensile strength, but the observed hardness values are very low, and at such low values considerable error is involved in converting hardness values to tensile strength. Small half inch thick samples were cut in each heat, and the surface ground on #400 grit paper. Brinell hardness test was performed and the results are given in Table No. 9. Rockwell B hardness values were also determined for annealed steel, and these values correspond well with the BHN obtained on the same samples.

Annealing

Samples cut from different heats were annealed at a temperature of 850° C for 2½ hours and then furnace cooled. The samples were kept in boxes without any protective atmosphere and the surface became oxidized during the annealing cycle. Metallographic samples were prepared for the purpose of examining the approximate carbon content of steel heats. The pearlite and ferrite areas indicated that carbon content of all heats was nearly equal to 0.1 to 0.15%. Photomicrographs for the annealed samples are shown in figures 3A to 3E.

Forging

Ingots from heats members 4, 5, 7, 10 and 17 were forged to a reduction of about 50%.

TABLE NO. 9
HARDNESS VALUES FOR STEEL

<u>Heat No.</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>	<u>13</u>	<u>14</u>	<u>15</u>	<u>16</u>	<u>17</u>
BHN As Cast	155	162	143	143	159	170	159	149	179	170	154	163
BHN As Annealed	112	131	114	121	121	116	121	124	126	-	-	-
RB (Annealed)	66	70	61	625	66	61	64	60	645	-	-	-

(Annealed at 850° C for 2½ hours and furnace cooled.)

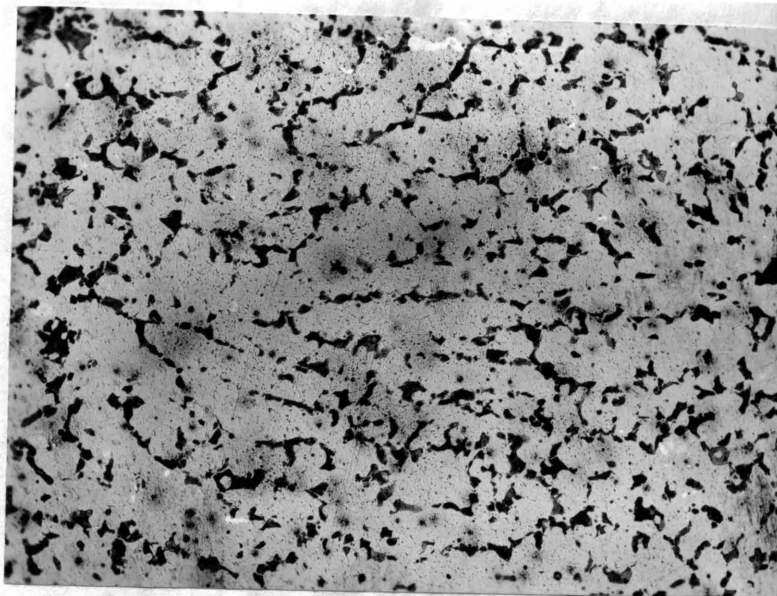


FIGURE 3A

HEAT NO. 9
ANNEALED, 3 HRS., 1650° F
100x
3% NITAL ETCH

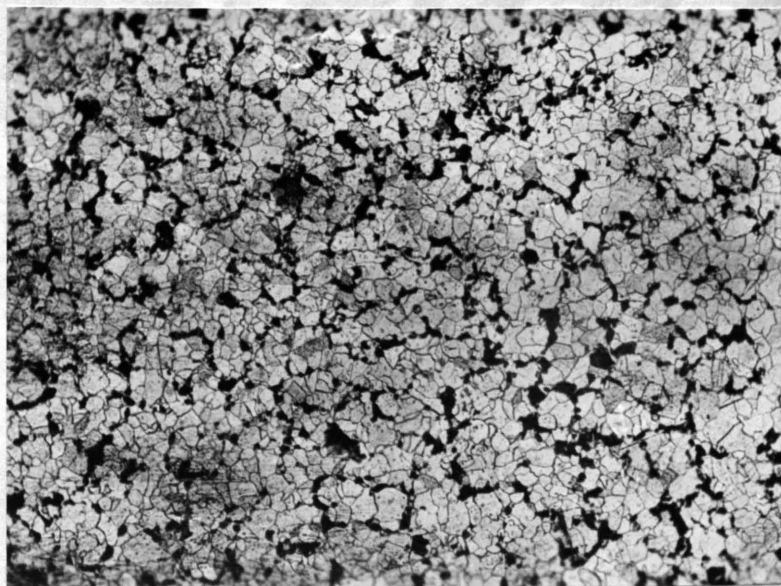


FIGURE 3B

HEAT NO. 10, ANNEALED
3 HRS., 1650° F
100x
3% NITAL ETCH

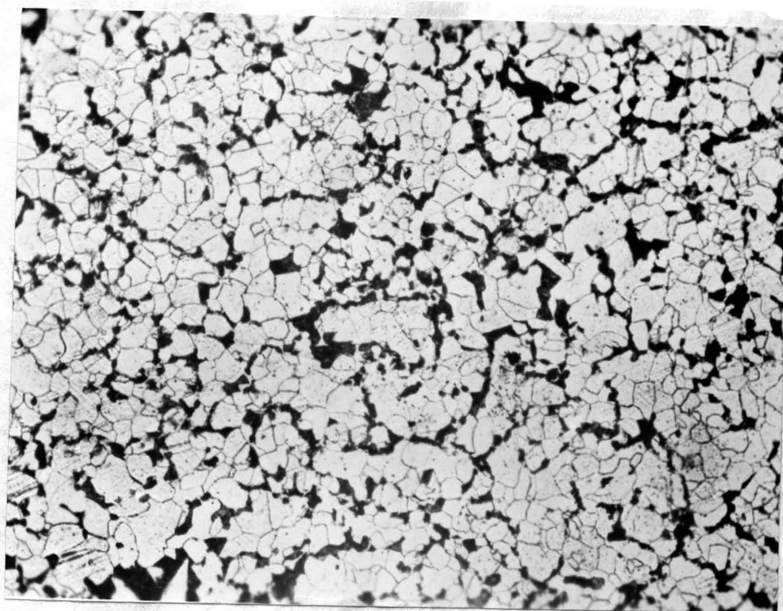
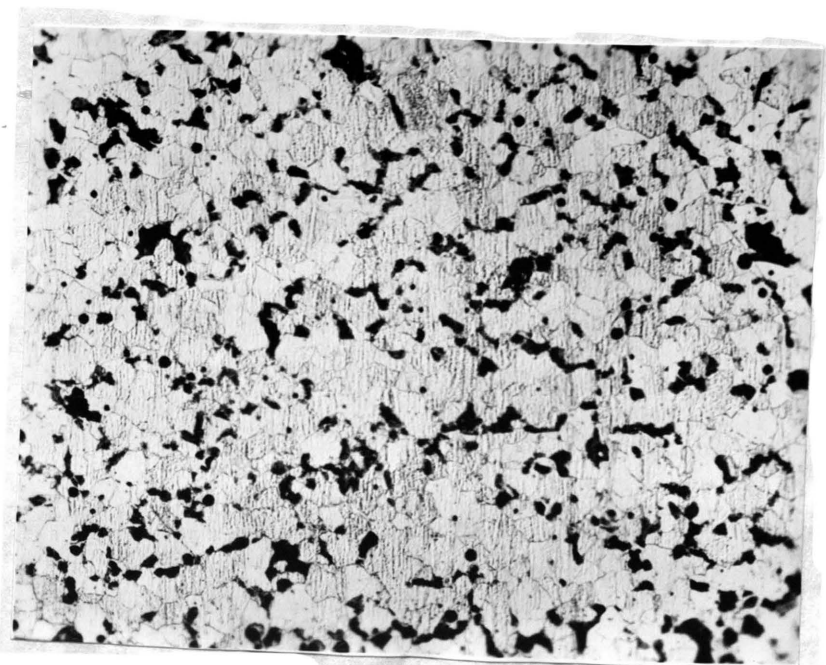


FIGURE 3C

HEAT NO. 11, ANNEALED 3 HRS.
AT 1650° F
100x
NITAL ETCH



FIGUR 3D

HEAT NO. 13, ANNEALED 3 HRS.
AT 1650° F
100x
NITAL ETCH

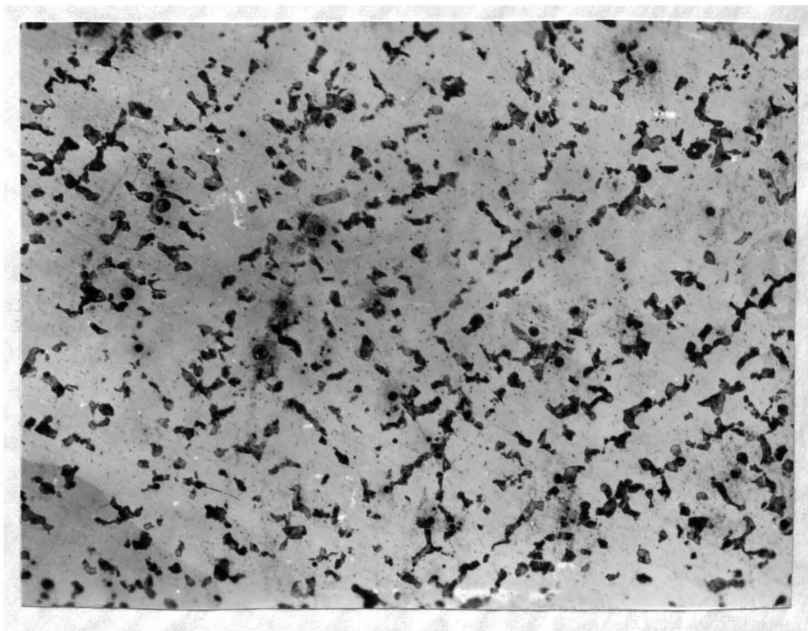


FIGURE 3E

HEAT NO. 14, ANNEALED 3 HRS.
AT 1650° F
100x
NITAL ETCH

The purpose of forging heats numbers 4 and 5 was to see if at a later stage it would be possible to forge ingots to smaller dimensions for tensile test pieces to be made from forged steel after rare earth additions. It was also proposed that hot forgeability may be assessed by forging pieces from the same temperature, but due to the temperature limitation of the forge furnace (2000° F), that could not be done.

Ingots number 7, 10 and 17 were forged to see if the shape of the inclusions change after forging. The reduction given was about 50%. This was done in three cycles for each sample. It has been observed in steels killed by Fe-Si and Fe-Mn that the MnS inclusions elongate on forging and rolling. From the properties of sulphides given in table number 4, it can be seen that rare earth sulphides are very stable and have high melting points. Also iron has no solubility for these sulphides at high temperatures while MnS is soluble in molten iron. These sulphides thus uniformly distribute themselves and do not segregate. The temperature at which forging was started was 2000° F, which is too low for the low carbon low alloy steel being investigated. Actual forging temperature for such a steel is 2350° F and finishing temperature 1950° F. The forged pieces were cooled in air after forging. It was found during forging that ingot from number 17 heat ruptured. This was probably due to low temperature.

The time given for heating the steel was 1½ hours. This included about 20 minutes of soaking the material at

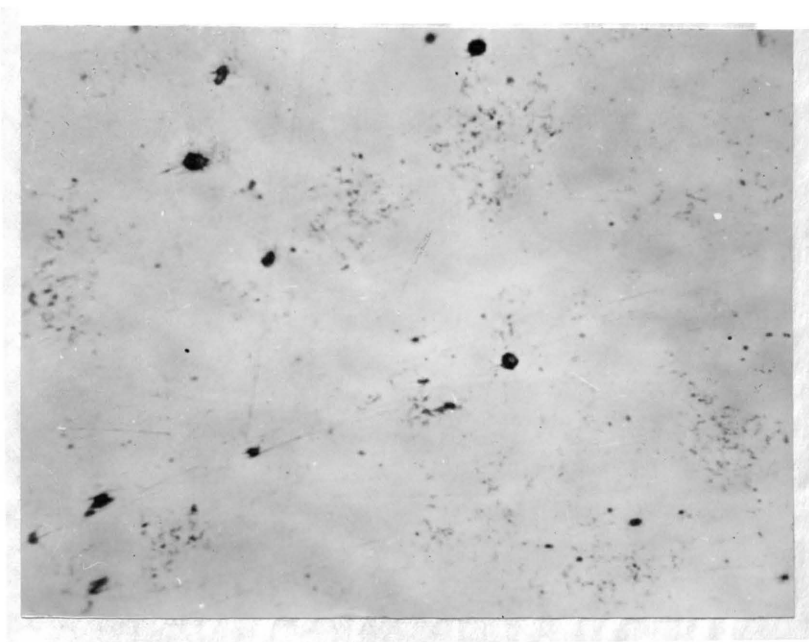


FIGURE 4A
SAMPLE NO. 7
500x
FORGED AT 2000° F
UNETCHED

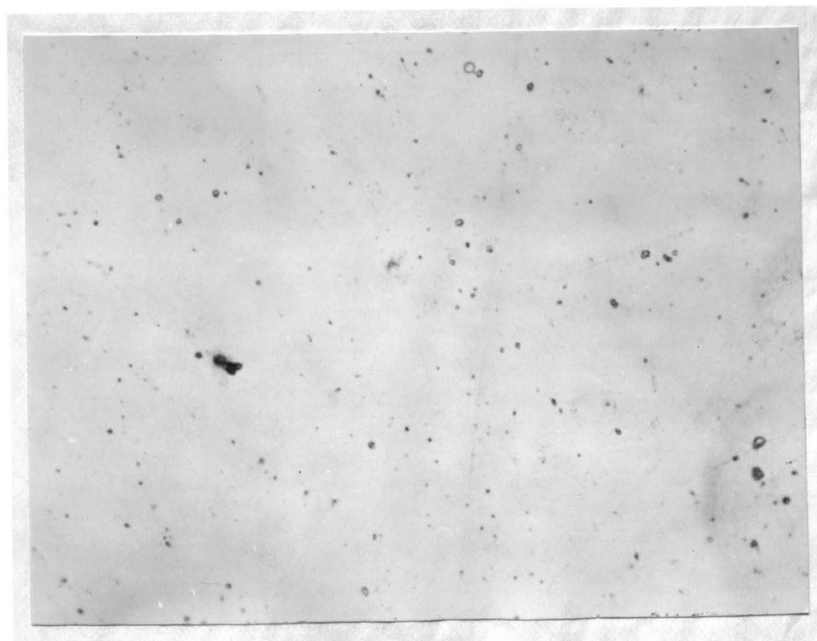


FIGURE 4B
SAMPLE NO. 10
500x
FORGED AT 2000° F
UNETCHED

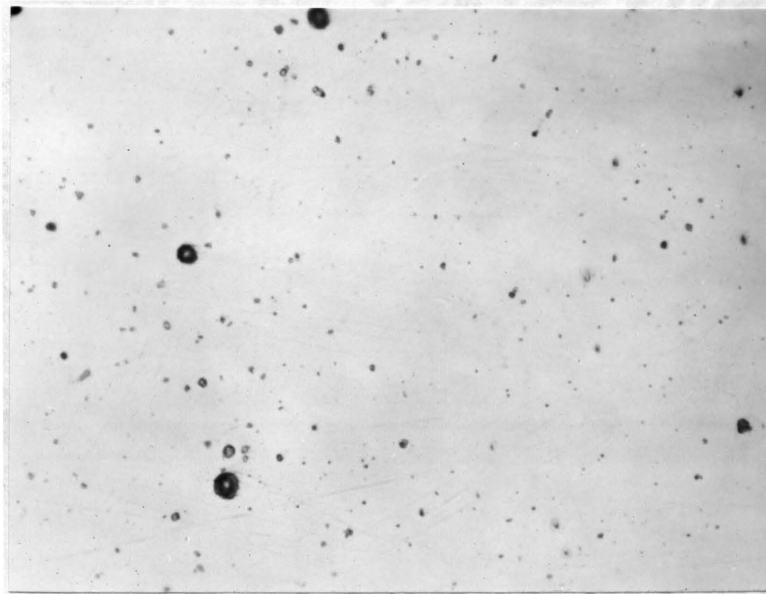


FIGURE 4C
SAMPLE NO. 17
500x
FORGED AT 2000° F
UNETCHED

at 2000° F. The time taken for the forge furnace to reach 2000° F was eight hours.

Three metallographic samples were cut, one each, along the length of the forged steels and these were polished, etched and micrographs obtained at a magnification of 500x. These are shown in Figures 4A, 4B and 4C.

Study of Inclusions

One of the criteria by which steel quality and fitness for some particular uses are judged is the content and character of nonmetallic inclusions. The very fact that they are nonmetallic and, therefore, incongruent with the metal lattice, has often been considered sufficient evidence of undesirability of large size inclusions. But in some steels, when they exist as fine inclusions, they are useful due to the fine austenitic grain size that results and the resistance that these finely dispersed particles offer to grain growth. Aluminum treated steel usually has a fine grain size. The refractory fine alumina particles act as nuclei and the resulting grain size is fine. The researches of Brophy²² and Mehl²³ show that aluminum, as dissolved in steel, imparts no significant effect upon grain size.

Inclusions are normally of two types; indigenous and exogenous. Indigenous inclusions have a fairly homogenous micro-distribution and any effect will be common throughout the steel. Exogenous inclusions are generally much larger and are often imperfectly "wetted" by the steel, both of which factors intensify their effects. The effect of indigenous inclusions on some of the properties are summarized below.

Machinability: When steel has a high sulphur content, machinability is considerably improved. The effect is explained on the basis of embrittling effect of sulphur. Oxide and silicate inclusions are considered harmful to machinability.

Grain Size: Alumina and aluminum nitride fine dispersion results in grain refinement. The high melting points of rare earth oxides indicate that their action may be similar to that of alumina.

Ductility and Impact Properties: These properties are affected directly by the quantity of inclusions present, but even more strongly on the size and distribution of the inclusions. Sulphide inclusions demonstrate that clearly. The size and distribution of sulphide inclusions can be varied by deoxidation practice. The effects are more evident on cast steels, but wrought steels also show difference in properties in longitudinal and transverse direction to the direction of rolling.

The sulphide inclusions are present either in globular form, when Si is used for deoxidation, or as part of eutectic when Al is used for deoxidation. It has been found that steels with globular sulphide inclusions give better impact properties than when present as a part of eutectic. High phosphorus content has also been found to affect the charpy values, and the effect of oxide inclusions is also presumed to be the same.

Brittle Fracture: The causes of brittle fracture in-

clude the formation of ferrite network or the massive carbide precipitation in the primary grain boundaries of the raw steel, as also the precipitation of inclusions in the primary grain boundaries, either during freezing or during slow cooling from a high temperature. MnS and AlN inclusions normally behave in this manner. They have appreciable solubility in gamma iron - a solubility that varies with temperature.

Hot Shortness: Sulphide inclusions are considered to be the cause of hot shortness.

Fatigue: The deleterious effect of nonmetallic inclusions on the fatigue endurance properties of steel is probably one of the most widely expected effect and yet experimental results show no such effect on endurance values from indigenous inclusions in their ordinary range of occurrence. But exogenous inclusions do have a detrimental effect on fatigue properties.

Weldability: Excessive sulphide inclusions have most deleterious effect on weldments, because of the characteristic of hot shortness imposed on the zone of fusion during solidification and cooling through the range of hot shortness. High sulphur free machining steels are difficult to weld. In metal for welding, sulphides are always undesirable.

Surface Appearance: Exogenous inclusions are mostly considered a cause of poor polish and pitting on stainless and low alloy steels, but such occurrences of agglomerated exogenous inclusions are rare.

In the present investigation metallographs have been taken for inclusion study from heats numbers 6 to 17 of as cast steels and for a few heats after annealing at 1650° C

for $2\frac{1}{2}$ hours. The results are shown in figures 5A to 5K.

Baumann Printing

The metallographic examination, though gives information about the size and distribution of inclusions, gives no information as to their constitution. Baumann prints were prepared for selected heats to see the type of sulphur distribution. The ingot pieces were ground first on coarse grit belt and finally on #400 grit grinding belt. The F-1 photographic printing paper was dipped in 2% H_2SO_4 for two minutes and was then held over the smooth ingot surface for two to three minute . It was then washed in water and then in hypo for ten minutes. The prints thus obtained showed the distribution of sulphur. In the case of ingots from number 9 and 14 heats, only faint brown spots appeared, indicating very little or no sulphur segregation. The Baumann prints for ingots from heats numbers 7, 9, 14, 16 and 17 are shown in figures 6A to 6E. The Baumann prints have been taken for all ingots from a section one inch above the bottom of the ingot.

Grain Size Determination

Austenitic grain size is recognized as an important property of steel, determining impact resistance, machinability, hardenability and creep strength. Work has been done to develop methods for disclosing austenitic grain size both in wrought and cast steels. In cast steels it is very difficult to obtain satisfactory results due to the complex nature of the structure. In most grain size determinations,



FIGURE 5A

HEAT NO. 67
250x
UNETCHED

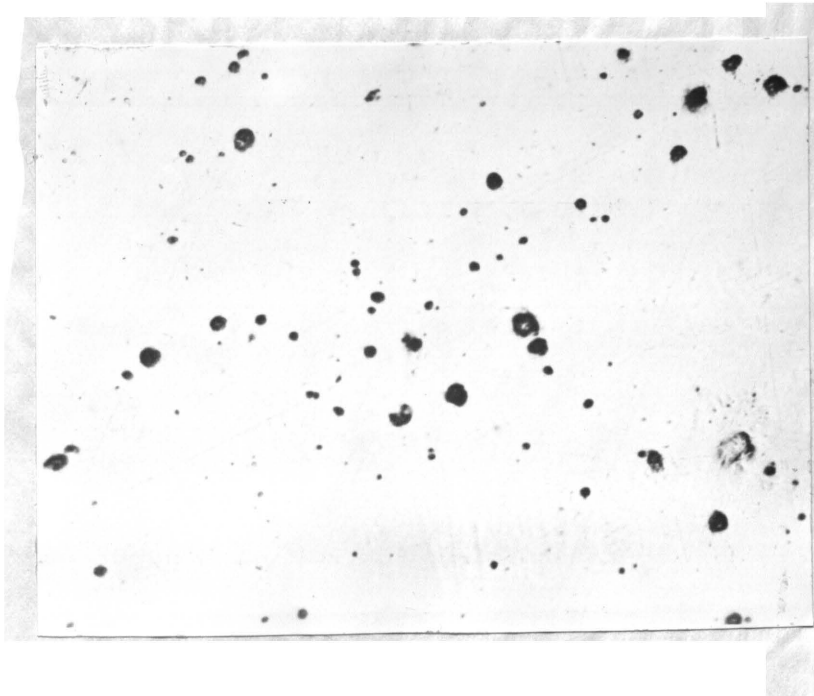


FIGURE 5B

HEAT NO. 76
250x
UNETCHED

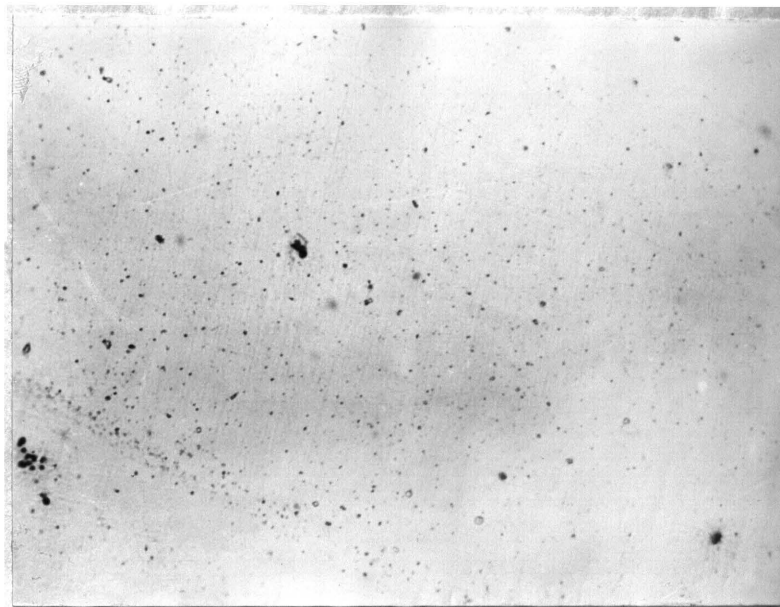


FIGURE 5C
HEAT NO. 9
250x
UNETCHED

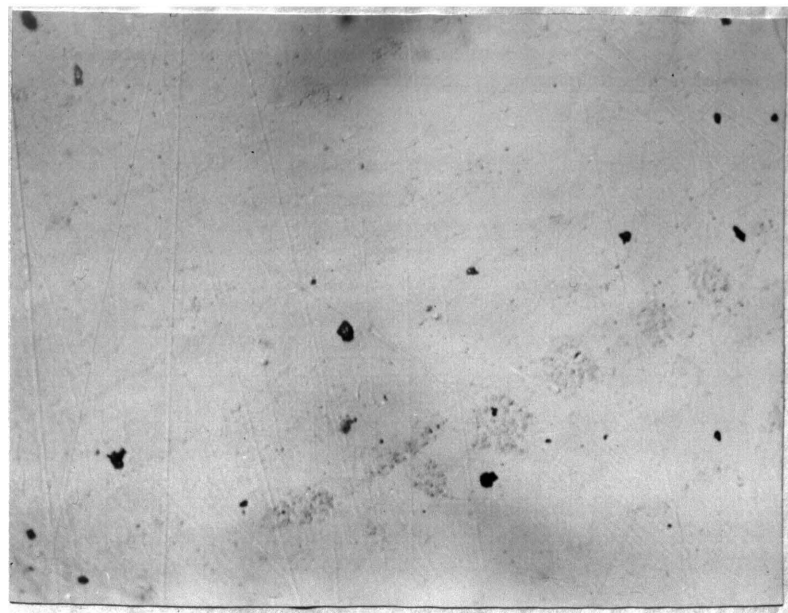


FIGURE 5D
HEAT NO. 10
250x
UNETCHED

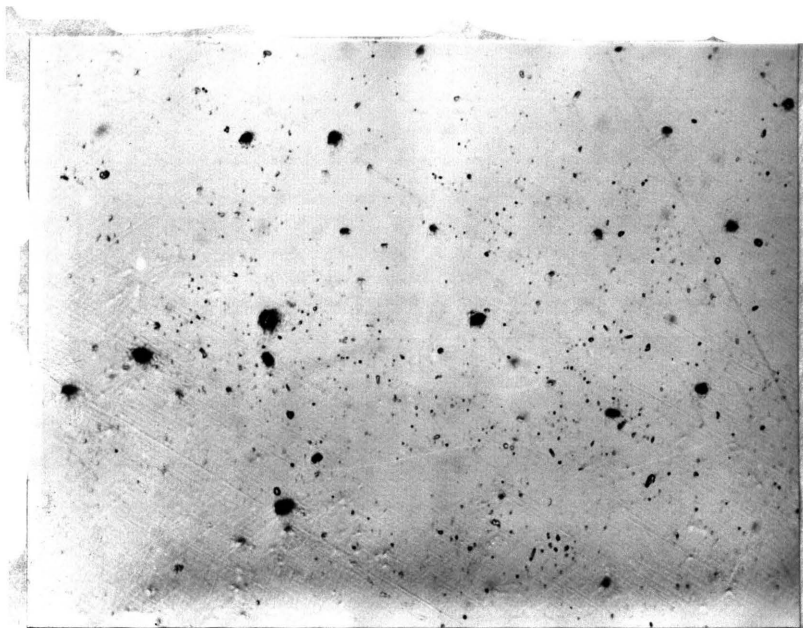


FIGURE 5E

HEAT NO. 11
250x
UNETCHED

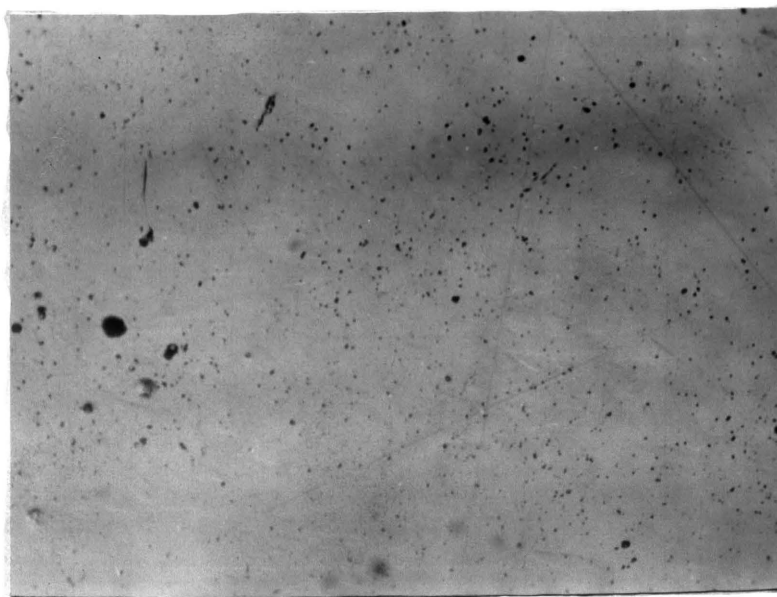


FIGURE 5F

HEAT NO. 12
250x
UNETCHED

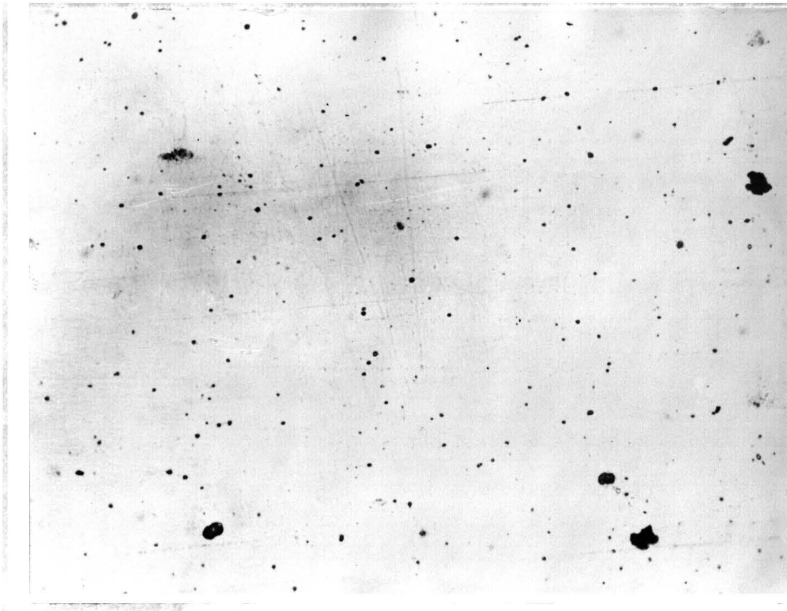


FIGURE 5G
HEAT NO. 13
250x
UNETCHED

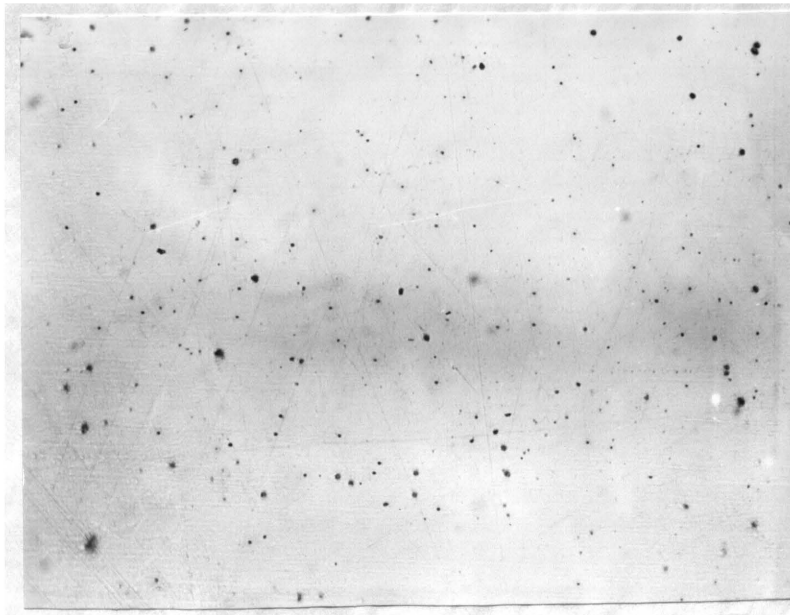
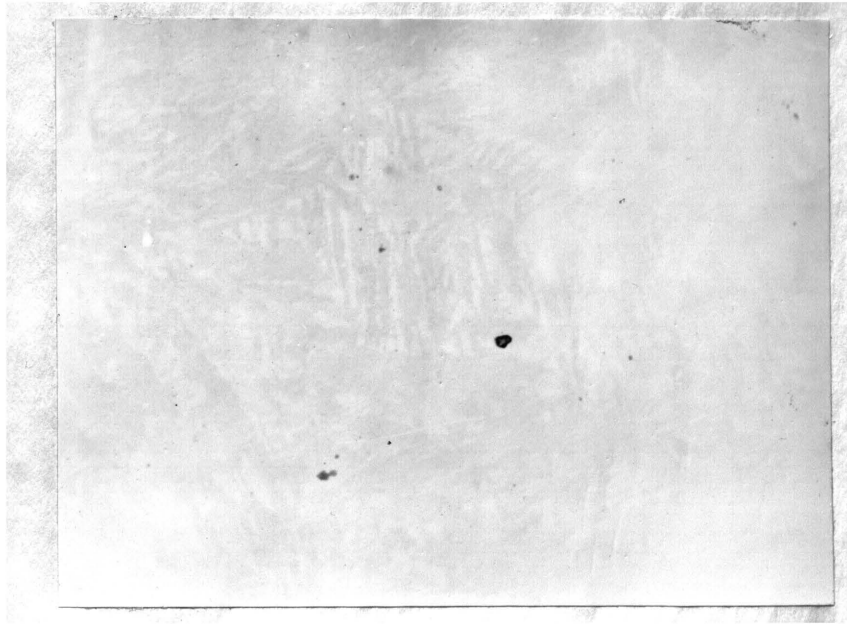


FIGURE 5H
HEAT NO. 14
250x
UNETCHED



HEAT NO. 15
250x
UNETCHED

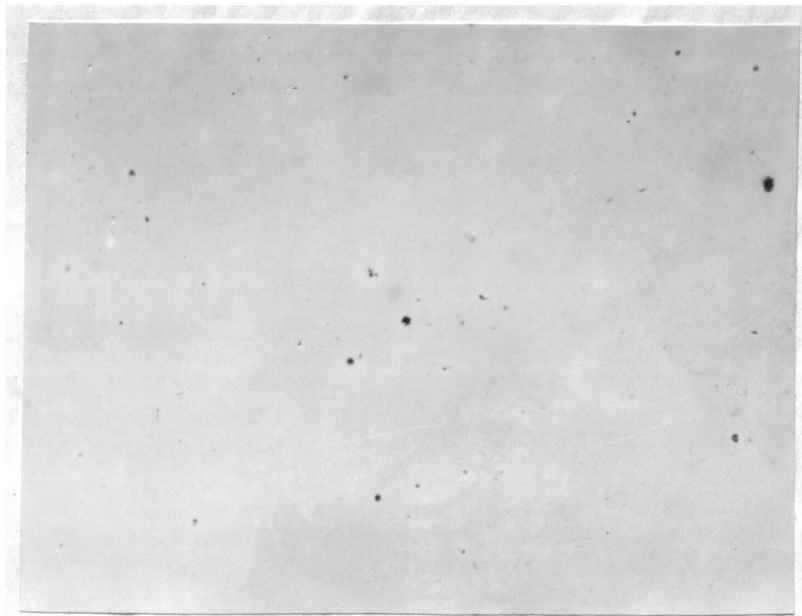


FIGURE 5J
HEAT NO. 16
250x
UNETCHED

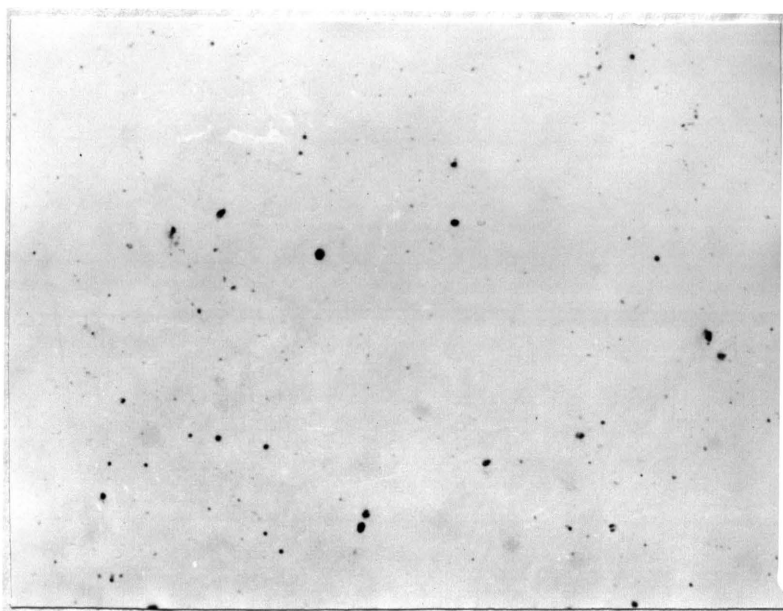


FIGURE 5K
HEAT NO. 17
250x
UNETCHED

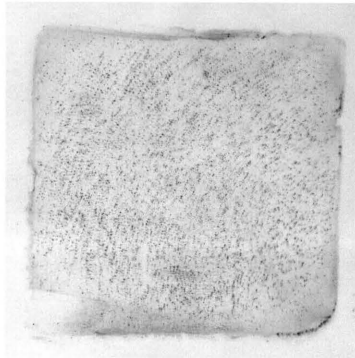


FIGURE 6A
BAUMANN PRINT
HEAT NO. 7

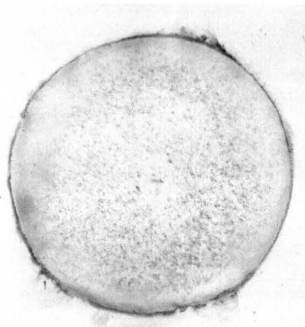


FIGURE 6B
BAUMANN PRINT
HEAT NO. 9

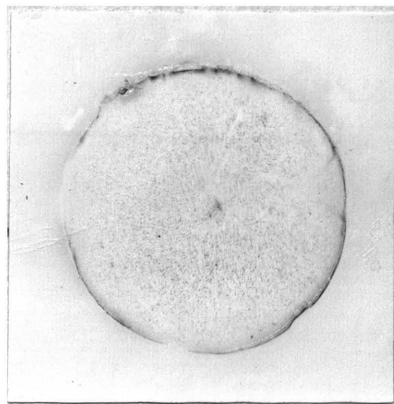


FIGURE 6C
BAUMANN PRINT
HEAT NO. 14

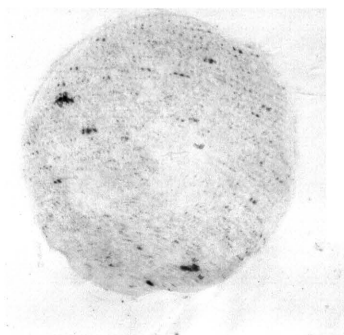


FIGURE 6D
BAUMANN PRINT
HEAT NO. 16

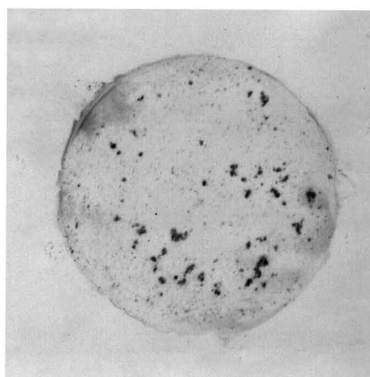


FIGURE 6E
BAUMANN PRINT
HEAT NO. 17

efforts are made to find the austenitic grain size, that no longer exists once the steel has been cooled to room temperature. In an excellent paper by Hawkes²⁴, methods for the determination of grain size have been discussed. Lohr²⁵ and Tisdale²⁶ have reported that grain refining is caused by rare earth additions.

In the present investigation, austenitic grain size has been determined on cast steels. The two methods, namely, "Martensitic etch method", and the "Mcquaid Ehn method", were applied for grain size determinations. Samples cut from the bottom portion of each ingot were used for the study.

Martensitic Etch Method: This method was developed by Vilella²⁷ to make possible the determination of previous austenitic grain size in quenched and tempered steel, but it has wide applications for grain size rating in general. The specimen to be rated is quenched after heating for one hour at 1700° F. The key operation in the test is etching the polished sample in a solution of one gram of picric acid and 5cc of concentrated HCl in ethyl alcohol. The steel samples in the present investigation were tempered after quenching at 600° F. The test depends on the fact that the former austenitic grain has a definite crystalline orientation differing from that of its neighbors, and that the orientation of martensite formed on quenching is fixed by the orientation of austenitic grains from which it comes. The rate of attack by the special etching solution varies greatly with the orientation of martensite, thus producing a typical "grain contrast etch". The grain size rating obtained by this method is given in Table No. 10; comparison being made with standard

ASTM grain size chart given in Metals Handbook²⁹.

Mcquaid Ehn Method: This method is most familiar of all the methods for obtaining a quantitative measurement of an austenitic grain size. It was developed by Mcquaid and Ehn²⁸ for the purpose of determining the size of austenitic grains in the surface layer of a case carburized low carbon steel. It is not much suited to high carbon steels. The standard test requires carburizing at 1700° F for eight hours and then slow cooling the sample to develop cementite, pearlite or pearlite-ferrite structure. In the present work, eight hours of carburizing at 1700° F gave a eutectoid case and pearlite-ferrite structure, as is shown in Figures 7A and 7B. The samples were pack carburized.

The carburizing time was extended to twelve hours in another run and that, too, gave a hypo-eutectoid case. The grain size rating was found by comparing with the standard ASTM chart²⁹.

Figures 8A to 8C show the structure after carburizing for twelve hours at 1700° F. The grain size ratings obtained by the above method for heats numbers 6 to 17 is given in Table No. 10.

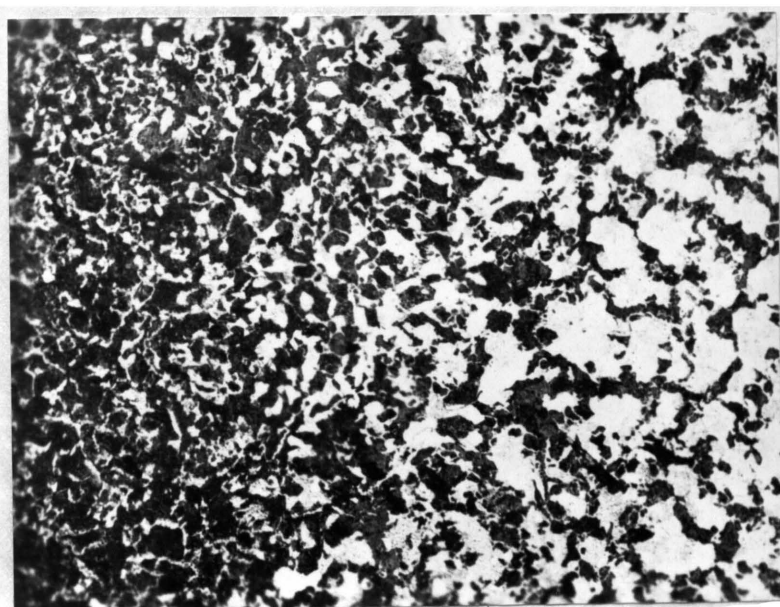


FIGURE 7A

HEAT NO. 10
CARBURIZED 8 HRS., 1700° F
100x
3% NITAL ETCH

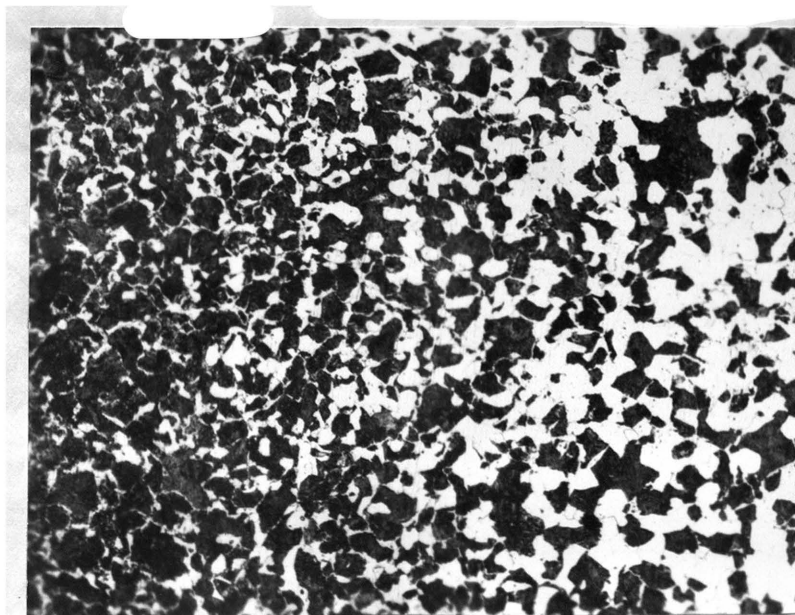


FIGURE 7B

HEAT NO. 15
CARBURIZED 8 HRS., 1700° F
100x
3% NITAL ETCHED

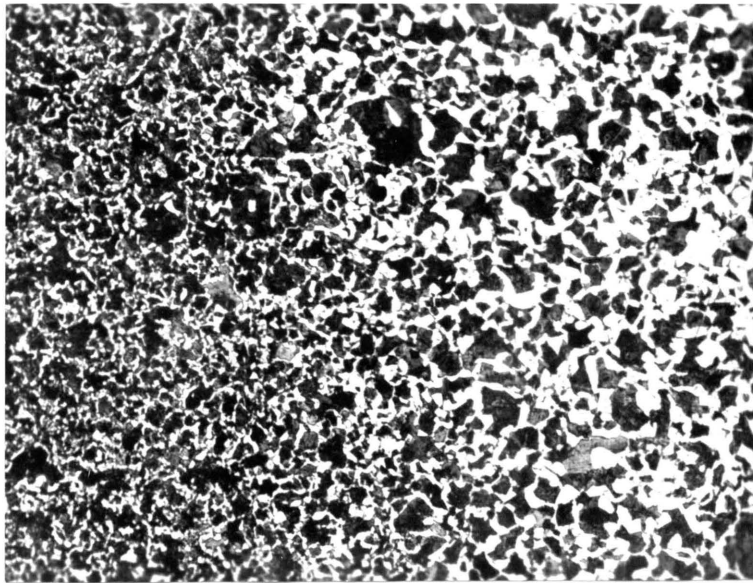


FIGURE 8A

HEAT NO. 15
CARBURIZED 12 HRS., 1700° F
3% NITAL ETCH
100x

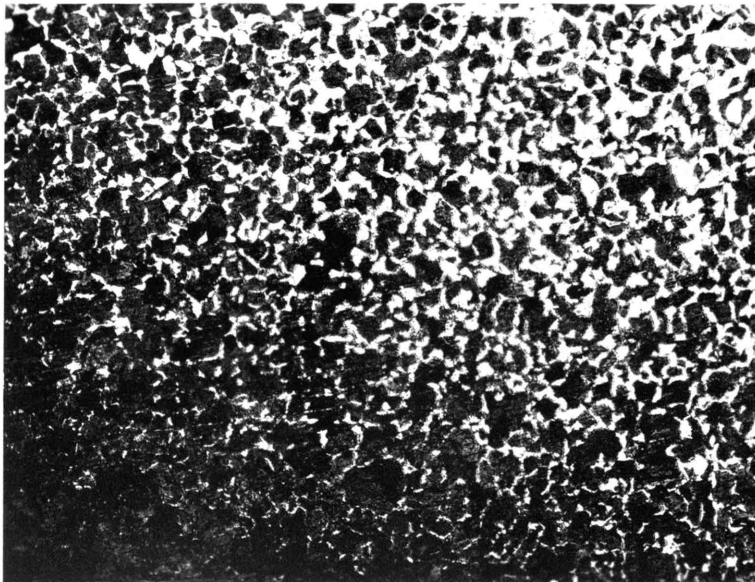


FIGURE 8B

HEAT NO. 16
CARBURIZED 12 HRS., 1700° F
3% NITAL ETCH
100x

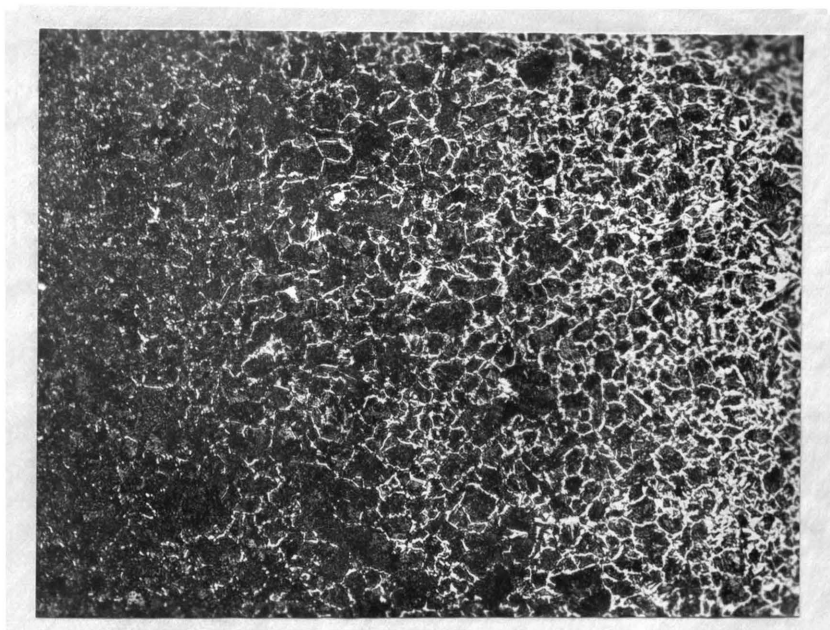


FIGURE 8C

HEAT NO. 17
CARBURIZED 12 HRS., 1700° F
100x
3% NITAL ETCH

TABLE NO. 10
GRAIN SIZE RATING

<u>Heat No.</u>	<u>Type of RE added</u>	<u>Martensitic Etch Method</u>	<u>Mcquaid-Ehn Method</u>
6	no addition	4-5	3-4
7	Oxide of Sm and Gd	5-6	6
8	Oxide of Sm and Gd	5-6	5-6
9	Oxide of Sm and Gd	6-7	6-7
10	Oxide of Sm and Gd	6-7	6-7
11	Fluorides of Rare Earths	2-3	3-4
12	Fluorides of Rare Earths	3-5	2-4
13	Fluorides of Rare Earths	3-4	2-4
14	Fluorides of Rare Earths	3-4	3-4
15	LanCerAmp No. 23	6-7	6-7
16	LanCerAmp No. 23	7-8	6-7
17	LanCerAmp No. 23	7-8	6-7

DISCUSSION OF RESULTS

Among the total of 17 heats produced by the use of high frequency induction furnace, the first five were experimental and details of their preparation are given in the appendix. The rare earth elements were added in latter heats by wrapping them in aluminum foil. With oxide additions, there was very little amount of rare earth oxide that could be dissolved. The ingots obtained for heats numbers 7 to 10 were clean at the surface. With rare earth fluoride additions, the ingot surface became dirty and slag was also entrapped at the surface, though its depth was not great. Similar effects were obtained in heats numbers 15 to 17 with addition of LanCer-Amp number 23. There was a slag layer around the inner surface of magnesite crucible which indicated that rare earth metals react with the crucible material. The chemical composition of steel heats is within tolerable limits with respect to different elements.

Desulphurization

Desulphurization studies made by Snellman⁸ indicated that in the small size crucible, induction melting practice, desulphurization was not satisfactory. In the case of open hearth and electric furnace practices, a 4-point sulphur drop has been reported by Lilliquist⁴, Russel and Knapp⁵. Reports on desulphurization³⁰ by rare earths have been published in "Steel"^{31, 32, 33}. From the analysis given in Table No. 8, it can be seen that the sulphur content of heat number 6, (without rare earth addition) is 0.019%. With oxide additions, a sulphur drop of 5 to 7 points has been obtained. The result is evident from the Baumann print shown in Figure 6B.

The rare earth fluorides seem to be less effective as a desulphurizer. The LanCerAmp, though, reduced the sulphur content, but this too was less effective than the rare earth oxides. In the case of heats numbers 7 to 10, increasing amounts of (Sm and Gd) oxides were added, and it appears that the sulphur drop was proportional to the amount of rare earth additions.

Figures number 6D and 6E further indicate that in numbers 16 and 17 heats, the sulphur is present, not with a dendritic pattern, but is distributed in grain form. In Figure 6A, the Baumann print shows dendritic fine distribution, with slight segregation in the center of the ingot, while no such segregation is noticed in heat number 17. The sulphur print obtained for heat number 9 does not show any appreciable spots. The sulphur drop obtained by rare earth metals can be explained on the basis of thermodynamic considerations. In the case of oxide addition, some complex phenomena is responsible for sulphur drop, which cannot be explained. It has been shown in Table No. 8 that with Sm and Gd oxide additions, there was no residual quantity of Sm or Gd detectable by spectrographic procedures. The amount of rare earths retained by the use of fluorides and LanCerAmp could not be found, though because of the excess quantity of rare earth additions, it is felt that there would be residual rare earths present in steel.

The hardness values for cast and annealed samples shown in Table No. 9 indicate a quick increase of hardness by small additions of rare earth elements, in any of the three forms.

The hardness then drops down, and with excess oxide addition, it is much lower than the hardness value obtained without rare earth additions.

The photomicrographs of annealed samples shown in Figures 3A to 3E show that with heats 9 and 14, there was still incomplete polygonization of structure. These figures helped in evaluating the carbon content of different heats in the initial stages, before chemical analysis arrangements were made.

The figures 4A, 4B and 4C show the inclusion distribution and form at 500x, after the material was forged to 50% reduction. The specimens were cut from near the surface, in a longitudinal direction after forging. It is shown that the form of inclusions is not much affected by forging in the case of number 10 and 17 heats. In ordinary steels, deoxidized with Mn, there is an elongation of MnS inclusions.

Inclusions

Figures number 5A to 5K show the distribution and shape of inclusions at 250x, of unetched samples, for the different heats. It appears that the rare earth additions considerably minimize the size of inclusions and also affect their form. Small amounts of rare earth additions are more effective in finely distributing the inclusions and with increasing additions, it is found that inclusions reappear and start becoming bigger also. This is shown in Figures 5D, 5H and 5K. Smaller additions of fluorides and oxides did not show much effect and about 0.7% of (Sm and Gd) oxides and 0.4% of rare

earth fluorides show very fine and regular distribution of inclusions.

In the case of LanCerAmp 23, very small addition of the alloy drastically modified the inclusions. The inclusions obtained were very fine, circular and the amount present was also less. This is shown in Figure 5I for heat number 15. Again, with increasing addition of LanCerAmp, the inclusions appeared as is shown in Figures 5I and 5K.

It seems that there is a certain optimum amount of rare earth addition that gives the best results as regards the shape, size and distribution of inclusions.

Grain Size

The results of grain size determination for different heats are given in Table No. 10. It has been found that 12 hours of pack carburizing treatment did not give a cementite network. The results indicate that the oxide additions and LanCerAmp additions refine the austenitic grain size of steel and the fluoride additions actually coarsen the grain size of steel. In the case of heats treated by fluoride, the grains were of mixed rating when observed by carburizing treatment, but the Martensitic etch method did not show much diversity in the grain size. The results of both the methods fairly correspond each other. It is found that maximum grain refinement occurs with LanCerAmp additions.

CONCLUSIONS

From the results obtained, it is possible to draw the following conclusions:

1. There is a possibility of desulphurization by rare earth additions in induction melted heats. Oxides of rare earths are more effective for desulphurization than fluorides and LanCerAmp.

2. The inclusions are affected by oxides, fluorides and LanCerAmp additions. Their distribution and size are considerably modified. Rare earths finely distribute the inclusions. Excessive rare earth additions cause inclusions to appear again.

3. Forging does not cause the inclusions to deform. They remain as a fine, spherical and regularly distributed phase even after forging.

4. Oxides and LanCerAmp appear to refine the grain size of the low alloy steel, while rare earth fluoride additions result in grain coarsening.

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APPENDIX

APPENDIX

PREPARATION OF HEATS NUMBER 1 TO 5

The purpose of making the initial five heats was to see whether or not the Armco iron could be satisfactorily melted and deoxidized. It was thought that metallographic examination of annealed samples may be used as an approximate guide as to the recovery of carbon used for recarburizing. The details of these heats are given here. Figure 9 shows the microstructure of Armco iron used for steel making in the form of punchings.

Heat Number 1: Armco iron was shot blasted and was melted in a magnesite crucible. The metal was poured into a square sand mold at a temperature of 3000° F. The time taken for the heat was about an hour. A sample cut at the bottom part of the ingot showed porosity and metallographic examination showed a dispersed dark etching oxide phase. Microstructure shown in Figure 10 indicated the oxide phase.

Heat Number 2: Weighed punchings of Armco iron were melted in magnesite crucible and deoxidation was done by dipping an aluminum rod into the melt. Fourteen grams of aluminum completely deoxidized 2000 grams of melt. The time taken for the heat was one hour, the tapping temperature was 3000° F. There was no porosity indicated by a polished bottom section of sand cast ingot. A sample was annealed at 1650° F for three hours and Figure 11 shows the annealed structure.

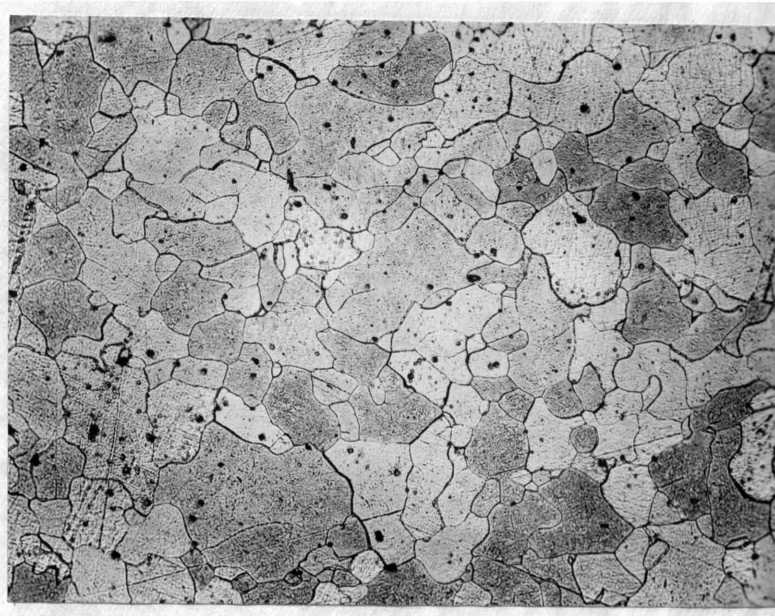


Figure 9

Armco Iron
100 x
3% Nital etch, 8 seconds

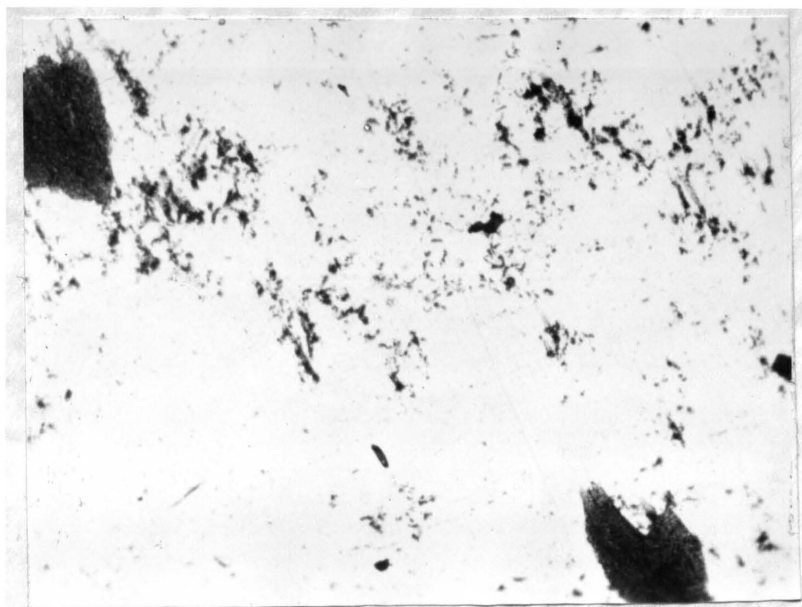


Figure 10

Heat No. 1
100 x
Unetched

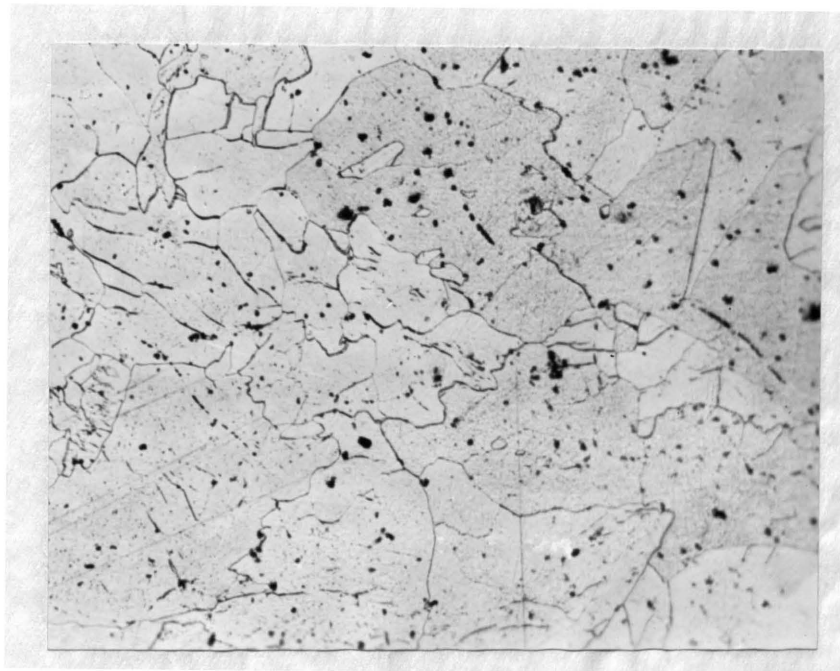


Figure 11

Heat No. 2
Annealed 1650° F, 3 hours

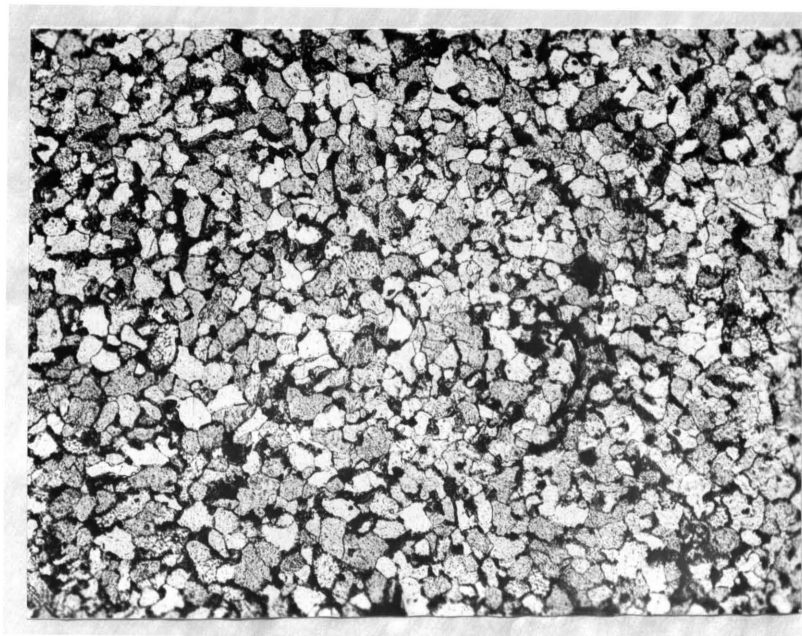


Figure 12

Heat No. 3
Annealed 2 hours, 1650° F, 100 x
3% nital etch

Heat Number 3: This heat was similar to number 2 heat, except that an effort was made to deoxidise the melt by the addition of excess graphitic carbon. The carbon in the end stages could not be dissolved properly. For 1800 gms of melt, 20 gms of carbon was used and the ingot was sand cast, the pouring temperature was 3000° F. A section at the bottom part of ingot showed porosity. A sample annealed for 2 hours at 1650° F showed pearlite and ferrite structure with 0.15-0.2% C. Figure 12 shows the microstructure of annealed sample.

Heat Number 4: This heat was prepared very carefully to judge the recovery of carbon. Magnesite crucible was used and the ingot was cast in sand mould. The heat was deoxidised with 10 gms of Silicon powder and 2.5 gms of electrolytic Mn. 25 gms of low-C ferrochrome and 15 gms of carbon were added about 10 minutes before pouring the heat. A sample from the bottom part of ingot was annealed at 1560° F for 1½ hours and Figure 13 shows the structure, indicating 0.5-0.6% carbon.

The remaining was forged from 2000° F and was reduced from initial 2½" x 2½" size to 1¼ in. dia. in 6 reheating cycles. Due to low forging temperature, the ends of the ingot cracked.

Heat Number 5: This heat was similar to number 4 heat except that the amount of C used was 15 gms. The amount of other additions for this heat are given in the representative log sheet prepared during making of the heat, as shown below.



Figure 13

Heat No. 4
Annealed 1560° F, 1½ hours
100 x
nital etch, 6 sec.

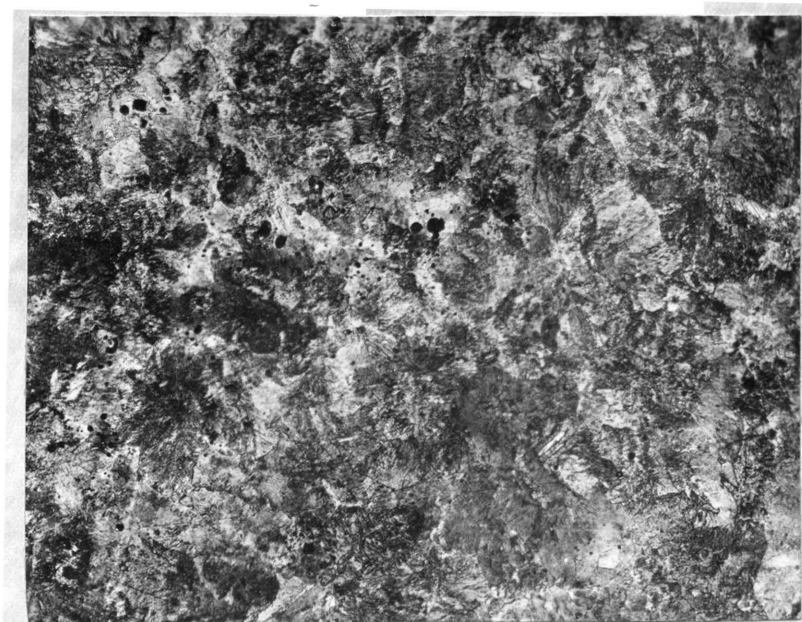


Figure 14

Heat No. 5
Annealed 1560° F, 1½ hours, 100 x
3% nital etch, 6 sec.

LOG SHEET HEAT NO. 5

Armco Iron punchings and welding rods taken	2000 gms
Silicon Powder added	10 gms
Electrolytic-Mn added	12 gms
Carbon	15 gms
Low Carbon Fe-Cr added	12 gms
Time furnace switched on	10-5 am
Pouring time	11-35 am
K W Rating	10
Pouring Temperature	2850° F
Weight of ingot	1365 gms
Remaining welding rod and punchings	463 gms
Crucible used	Magnesite
Ingot cast in sand mould	

A sample of this heat was annealed along with a sample from heat number 4 and Figure 14 shows the microstructure of the sample indicating nearly eutectoid composition.

Figure 15 shows an area with big size inclusions. These inclusions are angular.

This ingot was forged along with the ingot from number 4 heat and the section reduced to 7/8" dia. This ingot was also torn at the end due to heavy reduction and low forging temperature.

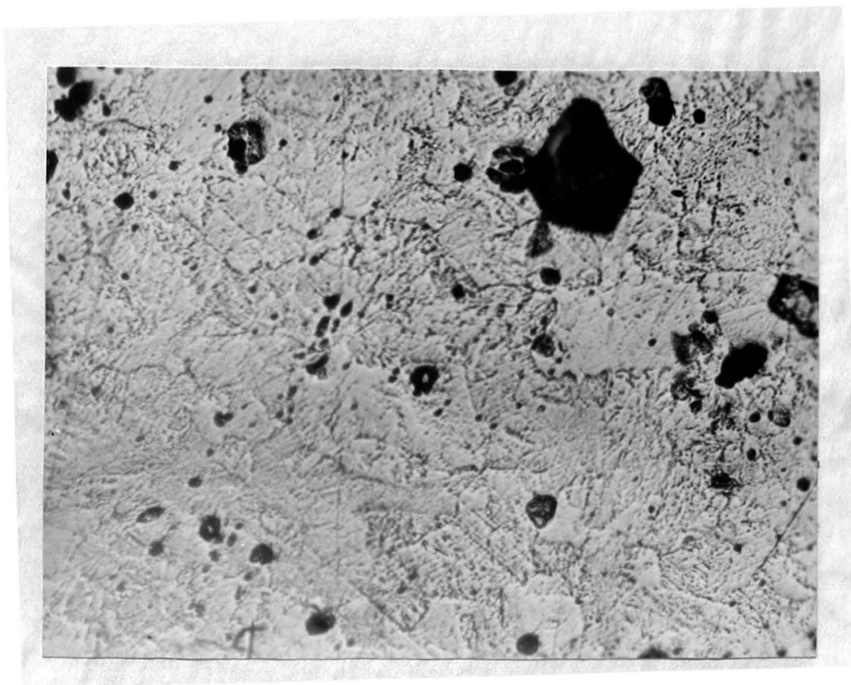


Figure 15

Heat No. 5
As cast 250 x
Nital etched and polished

SUGGESTIONS FOR FURTHER WORK

A few suggestion made by the oral examination committee members were as follows. It was observed that the study of inclusion and the effect on steel composition could be assessed better if the inclusions could be removed by electrolytic dissolution and their composition also determined.

The other proposal was to determine the composition of slag obtained after rare earth additions and also to study the phases present in slag by x-ray diffraction technique.

VITA

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