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IN GRAV  
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# **NIOBIMUM IN GRAY CAST IRON**

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**ABSTRACT** — *The potential for utilization of niobium in gray cast iron is appraised and reviewed. Experiments described in literature indicate that niobium provides structural refinement of the eutectic cells and also promotes pearlite formation. Consequently, niobium strengthens cast iron without too much increase in hardness. One of the demonstrated advantages of using niobium is that its addition to high carbon equivalent (CE) melts provides enough upgrading to allow their utilization in high quality castings with significant improvement in casting yield.*

## 1 - Introduction

### 1.1. Appraisal of Metallurgical Possibilities

Niobium has been considered as an alloying element for cast irons with a view to improve wear and/or strength. The usefulness of alloying cast irons with niobium depends essentially on its ability to dissolve in iron and/or react with other alloy or tramp elements commonly present in cast irons. Thus, niobium can be expected to interfere both with the solidification of and the solid state transformations in the alloy.

The maximum solubility of niobium in iron, in the liquid or in the solid state, is drastically reduced by the presence of carbon and/or nitrogen. Niobium combines with these elements forming carbonitrides, Nb(C,N), whose composition depends upon the carbon to nitrogen ratio<sup>1</sup>. These carbonitrides do not dissolve iron. Hence in a high carbon alloy such as cast iron Nb(C,N) would be essentially niobium carbide, NbC. These carbides can be formed in liquid at considerably high temperatures. From this point of view niobium would prevent iron carbide formation, promoting graphite. Recent calculations by Miyake and Okada<sup>2</sup> show that in Fe-3% C-Nb alloys, primary NbC precipitates well above the "liquidus temperature" of primary austenite. Moreover, the additional amount of carbide precipitated upon cooling to near "liquidus temperature" would be practically none. Consequently, upon reaching the temperature for austenite nucleation, particles of NbC are available for a possible interaction with that process.

In addition to combining with carbon and nitrogen, niobium reacts with oxygen and sulphur. These interactions are not so strong as observed in the case of titanium, however, they may be intense enough to affect graphite nucleation. Ueda<sup>3</sup> reported that by decreasing the nitrogen content in molten white iron, niobium promoted the formation of graphite nodules. On the other hand, niobium may also

affect the solidification as a carbide promoter (as opposed to graphitizer) as Mn does, being 50% less potent than chromium<sup>4</sup>.

According to Miyake and Okada's<sup>5</sup> calculations, the amount of niobium dissolved in liquid at 1100°C in an alloy with 3% C would be only  $4 \times 10^{-3}\%$ . Thus, not too much effect of niobium on the difference between Fe-C/Fe-Fe<sub>3</sub>C eutectic temperatures would be expected. However, "eutectic like" carbides are also observed in cast irons<sup>6</sup> which clearly indicates that niobium segregates to the liquid. This extra niobium may end up in "eutectic carbides".

The presence of niobium precipitated in liquid as carbonitrides may be important for allowing a refinement of the primary austenite dendrites through increased nucleation frequency as substrates. Since the graphite-austenite eutectic cells would form around the axis of the austenite dendrites<sup>6</sup> an obvious consequence of refining the austenite grains will be a refinement of the eutectic cells. This is important for providing high levels of strength to the material.

Finally, it is worth considering the possible effects of niobium on inoculation. According to one of current views<sup>7-9</sup> the active inoculant element (e.g. Si or Ca) forms metastable carbides which, upon decomposition, give free carbon to nucleate graphite. Therefore, the presence of strong carbide formers dissolved in the melt during inoculation may decrease the inoculant's power. In the case of niobium, however, this problem would not be very critical since at usual inoculation temperatures most of it should be combined as Nb(C,N) or NbC as suggested by the data of Fig. 1.

In summary, there are several possible routes for niobium to interfere with the solidification of cast iron. The alloy composition, the melting practice and the amount of niobium used will determine the dominant mode of niobium action. A quantitative description of this requires more fundamental studies.

As previously mentioned, the niobium remaining in solution in austenite after solidification can affect the properties of cast iron by interfering with the austenite decomposition, specifically preventing ferrite formation. The latter will be accomplished by the niobium eventually kept as a solute or precipitated in the solid state as a Nb(C,N). As a solute niobium can retard significantly the austenite to ferrite transformation<sup>8-10</sup> whereas by precipitating it would prevent carbon migration to the graphite flakes, thereby minimizing the formation of low carbon austenite zones which prompt ferrite formation.

As it will be seen later on, there are experimental data which support the contention that niobium may be a "pearlitizer", however, the specific mechanisms involved are not, at present, clear.

Summing up the qualitative analysis described in the foregoing, it appears that niobium has a considerable potential for being used as an alloying addition to cast irons. This point of view is supported in the next sections.

## **1.2. Literature Results**

Several authors <sup>5,10-15</sup> have investigated the influence of adding Nb to gray cast irons. The majority of these results indicates that niobium provides both increase in strength and refinement of the eutectic cells.

According to Campomanes and Goller <sup>15</sup> in addition to provide the effects already described, the use of niobium decreased the chill depth in melts with carbon equivalent (CE) close to 3.7%. This effect was obtained with and without using inoculation. These authors also reported a decrease in the size of the graphite flakes.

Castro et al. <sup>5</sup> have reported similar findings by adding up to 0.8% Nb to melts with 3.3% C - 2.2% Si and 0.65% Mn. Their results also indicated that the presence of niobium did not decrease the efficiency of inoculants. Ensor <sup>11</sup> investigated the influence of additions of niobium up to 3% also in hypo-eutectic melts (3.19/3.35% C - 2% Si - 0.035% Mn) and reported reductions in the amount and size of graphite as niobium content increased. Similar results were described by Sharan et al. <sup>12</sup>.

Among the authors cited <sup>5,10-15</sup> only Ueda's results <sup>11</sup> do not indicate structure refinement by niobium additions of up to 0.47% to melts with CE between 3.93 to 4.62% which is probably due to their naturally high tendency for graphite-austenite eutectic solidification.

In summary, these experimental results are not at variance with the analysis of the previous section. At present, however, it is unclear whether the action of niobium on the size of graphite flakes would be a direct or an indirect effect through austenite refinement as suggested by Sakamoto et al. <sup>6</sup> and by Lakeland <sup>18</sup>.

Niobium alloying has been cogitated for rolling mill rolls <sup>17-18</sup>. The results of Skobis et al. <sup>19</sup> indicate that in mottled-chromium-nickel cast irons, best characteristics for rolling mill applications were achieved with niobium contents between 0.03 to 0.06%. Interestingly, they also found that for Nb levels between 0.17 and 0.33% the amount of cementite decreased and a coarse dendritic structure was observed.

Some very recent results <sup>20</sup> indicate that the strengthening effect provided by niobium can be observed with CE values as high as 4.2%. This opens the possibility of using high CE alloys to obtain high casting yields and Nb alloying to achieve the high strength levels of high grade cast irons.

## **2 - Niobium Addition to Gray Cast Iron**

A literature review quickly showed that different methods for adding ferroniobium to the melts were used by the different researchers who also reported rather distinct extents of recovery.



Skobls et al.<sup>19</sup> added niobium (as ferroniobium) to the melt 5 to 7 minutes before pouring and obtained 50% recovery. On the other hand, Campomanes and Goller<sup>15</sup> achieved a recovery of almost 100% by using powdered (~ 100 mesh) ferroniobium and keeping the melt between 1500°C and 1600°C for 15 to 20 minutes. They also mentioned that a much lower recovery resulted from using coarse lumps (25 mm) of ferroniobium.

Castro et al.<sup>5</sup> used a graphite "bell" to introduce powdered ferroniobium into a melt at 1420°C. The bell was kept in the melt for 3 to 4 minutes. Recovery figures of 82 to 87% were achieved within 10 to 15 minutes with above practice.

Recent results<sup>20</sup> allow the evaluation of the recovery of ferroniobium (0.4 to 1 mm particle-size) addition to cast-iron. The experiments were conducted in an induction furnace (150 kg and 240 kVA) under four different conditions:

- a) *Addition to the Cold Charge ("COLD")*. Ferroniobium was added together with pig iron, steel scrap, and other ferroalloys. Superheating time was in the range of 5 to 30 minutes at 1470-1480°C.
- b) *Addition to the Hot Charge ("HOT")*. Ferroniobium and pig iron, scrap, and other ferroalloys were added together after melting 2/3 of the main charge. This procedure was designed to simulate the re-charge of an industrial furnace with superheating time of 5 to 30 minutes at 1470-1480°C.
- c) *Addition by Immersion ("BELL")*. Ferroniobium was added by means of a graphite bell which was kept in the melted charge for 10 to 15 minutes at 1470-1480°C.
- d) *Addition to the Melt ("MELT")*. Ferroniobium was added to the melt surface, at 1470-1480°C, after removal of the slag. 5 to 30 minutes were also used in this experiment.

The data of Table I indicate that the recovery rate in all cases is reasonably high (> 80%) and in the range usually obtained with other ferroalloys. Therefore for the conditions mentioned no special procedure is required to introduce ferroniobium in cast irons. However, it must be used in a finer form than usually employed in steel making.

### 3 - Niobium in High-Strength Gray Cast Irons

High strength cast irons can be made without resorting to special alloy additions, provide the carbon equivalent is kept sufficiently low. However, this route requires strict process control due to the undesirable mottling tendency of medium to low CE melts. The latter may entail machining problems due to uneven surface properties of the castings.

It is well known that by proper alloying medium to high CE cast iron, castings with reasonably high strength levels can be obtained. Moreover, these higher CE melts in addition to displaying a lower tendency to mottle also depict higher fluidity and less solidification shrinkage. Therefore, this route usually allows better casting yields.

The current understanding of the "modus operandi" of niobium in gray cast irons, summarized in section I, suggests that upgrading cast irons by alloying with niobium is a possibility worth considering. Therefore, the investigation described in this section was undertaken. The work consisted of characterizing the microstructure and mechanical properties of medium and high CE melts (Table II) subject to different degrees of inoculation with Ca-Si (30-35% Ca) or Fe-Si (60-85% Si)

**Table I — Chemical Analysis**

Addition Technique	Super Heating (min)	Chemical Composition (%)						Recovery (%) **
		C	Si	CE*	Mn	S	Nb	
"COLD"	5	3.1	1.98	3.76	ND	ND	0.71	95
	20	ND	ND	—	ND	ND	0.69	92
	30	3.05	2.05	3.73	0.38	0.095	0.70	93
"HOT"	5	3.05	1.97	3.70	ND	ND	0.69	89
	20	ND	ND	—	ND	ND	0.68	85
	30	3.0	1.97	3.66	0.36	0.092	0.69	89
"BELL"	5	2.98	1.98	3.64	ND	ND	1.31	83
	20	ND	ND	—	ND	ND	1.34	87
	30	2.99	1.98	3.64	0.98	0.093	1.32	84
"MELT"	5	3.15	1.94	3.8	0.40	0.090	1.26	+ + 84
	20	ND	ND	ND	ND	ND	1.31	+ + 87
	30	3.10	1.96	3.75	0.41	0.090	1.28	+ + 85

(\*) CE = %C + 1/3%Si

ND — not determined

(\*\*) for additions of 1.154% Fe-Nb

(+ +) for additions of 2.31% Fe-Nb

**Table II — Chemical Composition of Castings (%)**

	C	Si	CE*	Mn	S	P	Nb
I	3.6-3.8	1.41-1.67	4.1-4.3	0.36-0.42	0.053-0.070		
II	3.11-3.24	1.76-2.01	3.68-3.90	0.27-0.40	0.047-0.068	0.070	0.25;0.75;1.5

(\*) CE = %C + 1/3%Si

I = High CE in the text

II = Medium CE in the text

**Table III — Metallographic Data-Medium CE Melts  
Inoculated with Ca-Si**

Ca-Si (%)	Nb * (%)	Ferrite (%)	Graphite — "A"	
			(%)	Size-ASTM
0.2	—	2	80	4/5
	0.25	2	90	4
	—	2	100	4
	0.75	2	100	4
	—	—	100	4
0.4	1.5	—	100	5
	—	—	100	4
	0.25	—	100	4
	—	—	100	4
	0.75	—	100	4/5
0.6	—	—	100	4
	0.25	2	100	4
	—	—	100	4
	0.75	2	100	4/5
	—	2	100	4
	1.5	—	100	4/5

(\*) Nominal

**Table IV — Metallographic Data-Medium CE Melts  
Inoculated with Fe-Si**

Fe-Si (%)	Nb * (%)	Ferrite (%)	Graphite — "A"	
			(%)	Size-ASTM
0.2	—	10	50	4/5
	0.25	5	30	5
	—	5	60	4
	0.75	—	70	4
	—	5	70	4/5
0.4	1.5	—	50	5
	—	2	80	4
	0.25	—	90	4
	—	5	80	4
	0.75	5	70	4/5
0.6	—	2	80	4/5
	1.5	—	60	4/5
	—	5	80	4
	0.25	3	90	4/5
	—	5	90	4
0.6	0.75	5	80	4/5
	—	2	80	4
	1.5	—	80	4/5

(\*) Nominal

and alloyed with niobium up to 1.5%. These melts were prepared in an induction furnace and ferroniobium was added to the melt top as mentioned in section 2. For each experimental run there was an unalloyed melt for comparison. Tensile tests and metallographic examination were performed on 30 mm diameter cast cylinder. Chill tests were carried on after the ASTM standard A-367-64.

### *3.1. Mechanical Properties*

The data of Fig. 2 show that niobium adds significantly to the strength of both medium and high CE melts. Class upgrading is a clear possibility.

The strengthening benefit of alloying with niobium is obtained whether the melt is treated with Fe-Si or with the more efficient inoculant, Ca-Si. The latter, however, entails higher tensile strength. The results of Fig. 2 also suggest that, for a given increase in strength, the amount of niobium required depends upon the extent of inoculation. Although no efforts were made to optimize composition, the results indicate that an intermediate level of inoculation (~ 0.4%) allows niobium addition to be in the range of 0.25% which is reasonable because, in this case, only a small quantity of niobium carbides or carbonitrides is obtained in the microstructure.

The hardness of the castings was found to increase with the addition of niobium (Fig. 3). However this increase was neither too severe nor too sensitive to the amount of niobium added.

Therefore, from an application point of view, niobium additions in the range of 0.25% are sufficient to provide 10 to 30% strengthening to the considered irons without too much an increase in hardness. This benefit may be enough to upgrade castings which otherwise do not meet the specification of higher strength classes.

### *3.2. Microstructure Evaluation*

Both medium and high CE casting were subjected to optical metallography investigation.

Niobium was found to refine the eutectic cells (Fig. 4). The effect is additive to that of the standard inoculants (Fe-Si or Ca-Si) and is observed in high and medium CE melts. The data of Fig. 5 indicate that, for a given extent of inoculation, the eutectic cell-size of Nb alloyed product is proportional but finer than observed in the control castings made without Nb. However, the data did not allow that effect to be precisely quantified. On the other hand, it is clear that by increasing the amount of inoculant (Fe-Si or Ca-Si) from 0.2 to 0.6%, the additional refinement provided by niobium decreased from about 20%

**Table V — Metallographic Data-High CE Melts  
Inoculated with Fe-Si**

Fe-Si (%)	Nb * (%)	Ferrite (%)	Graphite — "A"	
			(%)	Size-ASTM
0.2	—	10	80	3
	0.25	5	70	3/4
	—	5	70	4
	0.75	5	70	4
	—	2	80	3
	1.5	—	80	4
0.4	—	2	80	3/4
	0.25	2	80	4
	—	2	70	3
	0.75	2	70	3/4
	—	2	80	3
	1.5	—	80	3/4
0.6	—	15	100	3
	0.75	10	100	3/4
	—	2	80	3
	0.75	2	80	3/4
	—	5	100	3
	1.5	2	100	4

(\*) Nominal

**Table VI — Metallographic Data-High CE Melts  
Inoculated with Ca-Si**

Ca-Si (*) (%)	Nb (*) (%)	Ferrite (%)	Graphite — "A"	
			(%)	Size-ASTM
0.2	—	5	90	3
	0.25	2	100	3/4
	—	2	80	3
	0.75	2	100	4
	—	2	80	3
	1.5	—	100	4
0.4	—	—	100	3/4
	0.25	—	100	4
	—	2	100	3
	0.75	2	100	3/4
	—	2	100	3
	1.5	—	100	3/4
0.6	—	2	100	3
	0.25	2	100	3/4
	—	2	100	3
	0.75	2	100	3
	—	10	90	3
	1.5	2	100	4

(\*) Nominal

to 6%. This effect, which should not be confused with "fadding" merely reflects the fact that standard inoculants efficiently promote the graphite nucleation. Actually, the observation of Nb(C,N) particles inside the austenite dendrites<sup>5</sup> and Fig. 6, supports the contention that the ability of niobium to refine the microstructure of the casting derives from the catalytic action of Nb(C,N) particles on austenite nucleation. Thus, the refinement of the eutectic cells by niobium would be an indirect process and, as such, much less effective than achievable with inoculants (viz. Fe-Si or Ca-Si) which directly interfere with graphite nucleation.

Niobium (from 0.25 to 1.5%) was found to increase the chill depth of both high and medium CE melts, Fig. 7. The effect was found, as should be expected, to decrease with increasing the extent of inoculation with Fe-Si or Ca-Si. Again no specific trend between chill-depth and niobium content could be readily identified. This tendency of niobium to promote chilling is at variance with the results of refs. <sup>5,15</sup>. However, it agrees with data reported by Merchant<sup>4</sup>. The reasons for this discrepancy are not clear at present.

Additional data pertaining of ferrite and type-A graphite are given in Tables III to VI. These data indicate that neither the percentage of type-A graphite nor that of ferrite are significantly altered by niobium. On the other hand the size of type-A graphite flakes tends to be smaller in the niobium-alloyed melts. This suggests that niobium, per se, does not promote graphite formation although it can interfere with the graphitization process by decreasing the size of the eutectic cells as shown in the foregoing.

### *3.3. Microstructure/Properties Correlation*

It is beyond the scope of this work to discuss the details of microstructure-properties relation in gray cast irons. For the present purpose it suffices to say that the picture which emerged from the metallographic analysis correlated well with the change in mechanical strength and hardness promoted by adding niobium to the experimental melts. It is proposed that up-grading resulted from the refinement of the eutectic cells and concurrent decrease of flake size. Of course, the reduction in ferrite with the consequent increase in pearlite content may be an additional bonus.

## **4 - Practical Developments**

In addition to the metallurgical benefits discussed in the foregoing, the use of niobium when properly administrated can entail production

cost benefits. Improvement of casting yield by reduction of risers is a possibility when niobium alloying is considered.

Reduction or elimination of risers in gray cast iron castings can be accomplished by allowing a higher carbon-equivalent and a lower Si/C ratio to obtain about 2.4% eutectic graphite. However, this procedure entails a loss in tensile strength which, sometimes, cannot be accepted. Alloying can balance this loss in strength and present results indicates that niobium is particularly effective in doing that.

The example selected merely to demonstrate the above was the casting of brake drums with 250 MPa (min) gray cast iron. This component is usually cast with a hypoeutectic iron (with 2.25 - 2.30% eutectic graphite) and requires great risers to obtain proper soundness. In the specific case considered here, elimination of the latter would allow an increase in casting yield from 57.2 to 69.3%.

Brake drums cast with and without risers are shown in Fig. 8. The latter cast with a 4.2% carbon equivalent melt (3.65% C, 1.78% Si, 0.29% Mn, 0.47% Nb) was inoculated with 0.4% Ca-Si. These experimental castings exhibited excellent soundness and a tensile strength (determined from 30 mm diameter specimens) of 267 MPa which is above the specified strength level. According to the data of Fig. 2 inoculation with Fe-Si could have been applied. However, in this case, probably higher levels of niobium would be required to meet the 250 MPa strength level.

Attempts to use the more traditional additives (Cu, Cr and Sn) to upgrade the tensile strength of the higher carbon equivalent heats did not succeed in providing the necessary 250 MPa level. The composition used in these trials was in the following range: 3.55 - 3.64% C, 1.41 - 1.44% Si, 0.25 - 0.35% Mn, 0.14 - 0.16% Cr, 0.08 - 0.10% Sr, 1% Cu and 0.05% P. These melts contained between 2.40 and 2.49% eutectic graphite and displayed tensile strengths from 218 to 234 MPa when inoculated with Fe-Si and 225 to 241 MPa if inoculated with Ca-Si.

Fig. 9 shows a cylinder sleeve currently produced by Fundação Tupy for the Honda 125 motorcycle (\*). The sleeve is made of high quality gray cast iron with adequate P content and niobium added in the range of 0.1 - 0.8%. The scanning electron photomicrographs of Fig. 10 is typical of the material. The area inside the dashed square in Fig. 10(a) is shown in greater details in Fig. 10 (b). The constituents marked 1, 2 and 3 were identified by EDX as predominantly NbC, manganese sulfide and iron phosphide, respectively. The material was found to exhibit good thermal conductivity and resistance to both wear and thermal shock. Moreover, the parts display excellent homogeneity and mechanical strength which decisively contribute to the performance and durability of the Honda engines.

(\*) Figure and data presented by permission.

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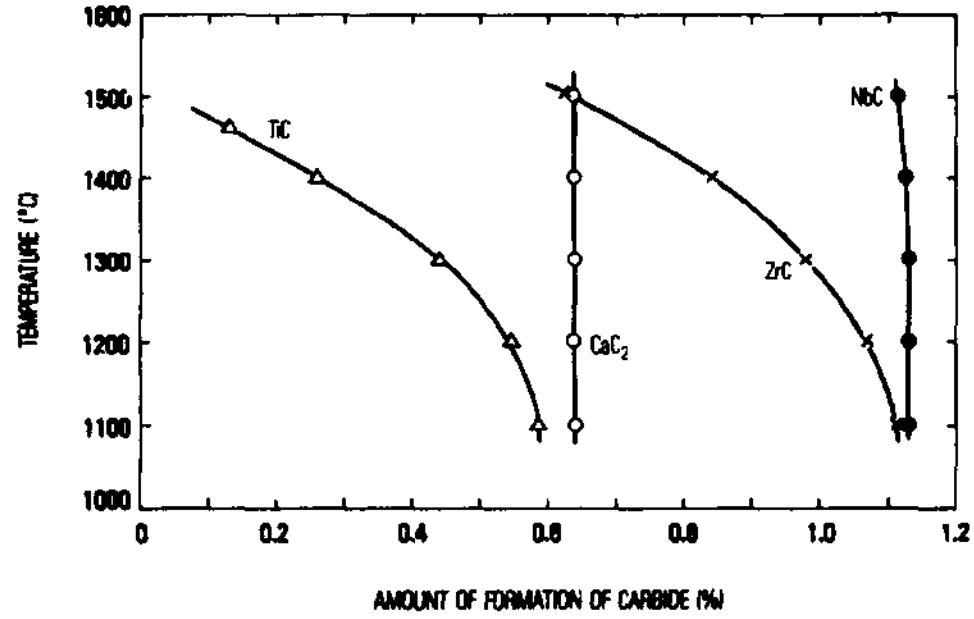


Fig. 1 — Variation in the amount of carbides precipitated in Fe-3% C - x (x = 0.4% Ca; 1% Nb; 0.5% Ti and 1% Zr). Notice that in every case x at% = 0.5. (2)

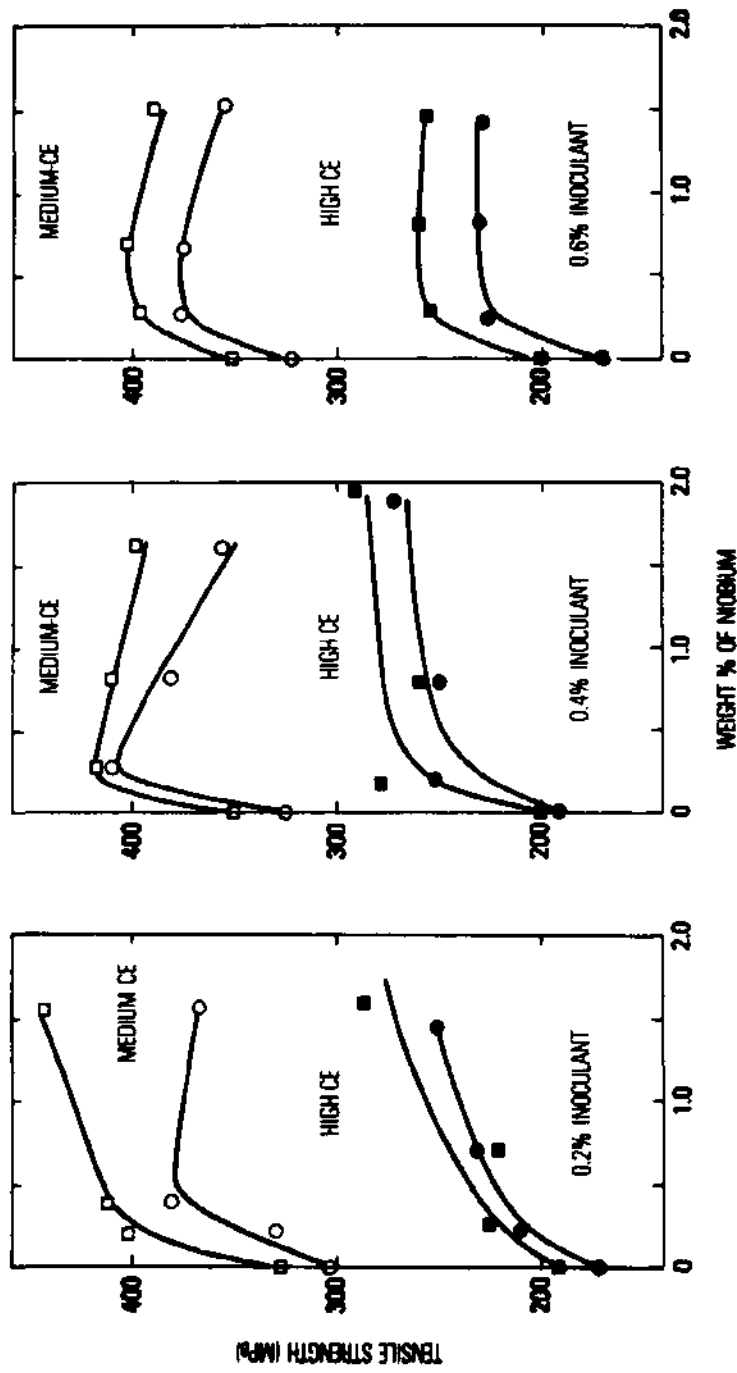


Fig. 2 — Variation of the mechanical strength of medium and high CE gray cast irons as a function of niobium content at different inoculation extent with Ca-Si (■ □) or Fe-Si (● ○).

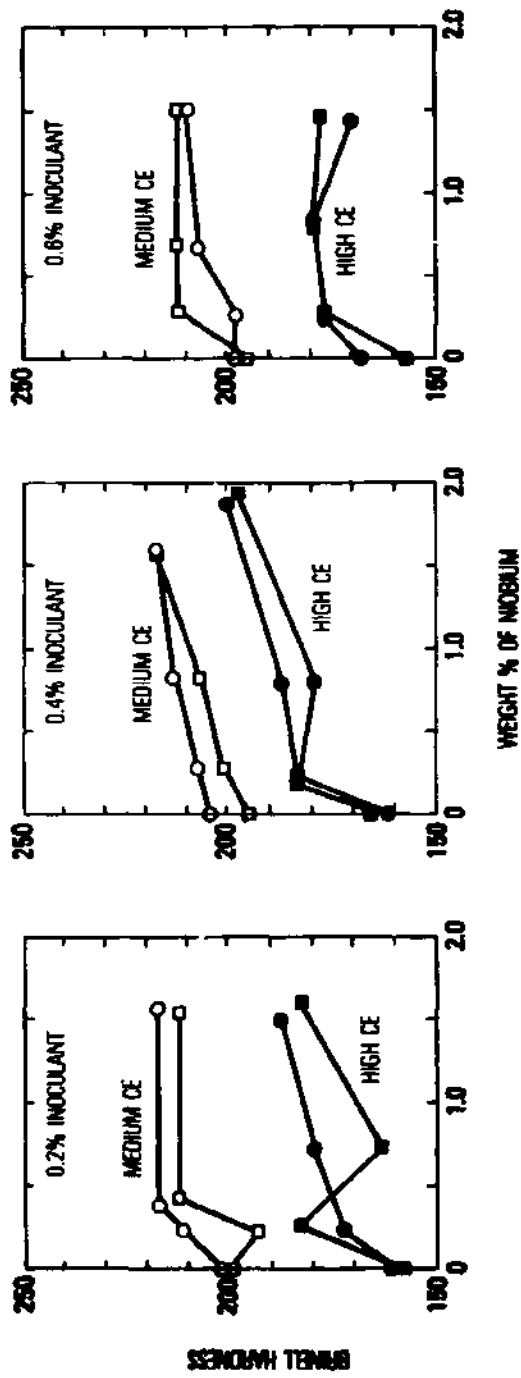


Fig. 3 — Variation of the hardness of medium and high CE gray cast irons as a function of niobium content at different inoculation extent with Ca-Si (□, □) or Fe-Si (●, ●).

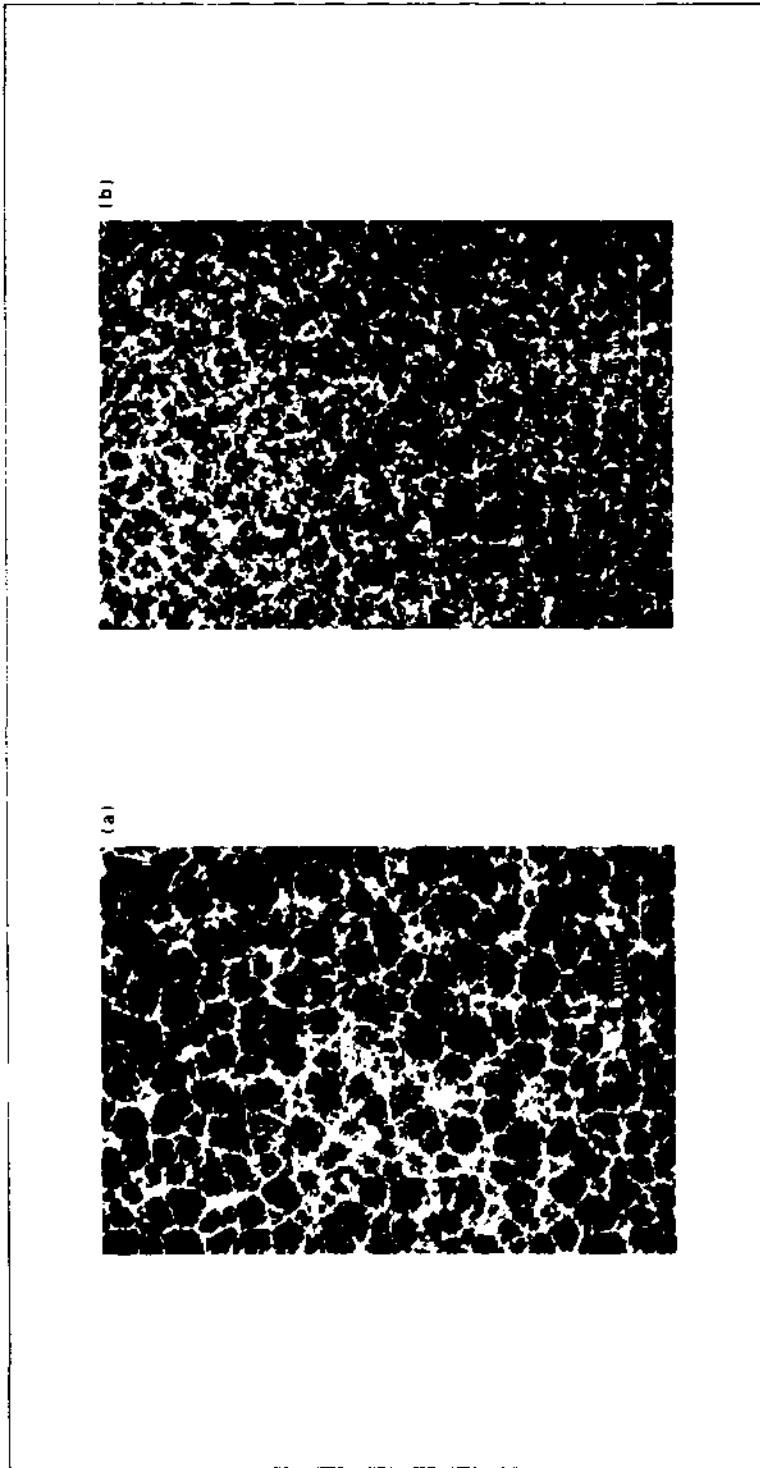


Fig. 4 — Photomicrographs. Eutectic cells in high CE cast iron. (a) Inoculated with 0.4% Fe-Si and alloyed with 0.9% Nb. (b) Inoculated with 0.4% Fe-Si.

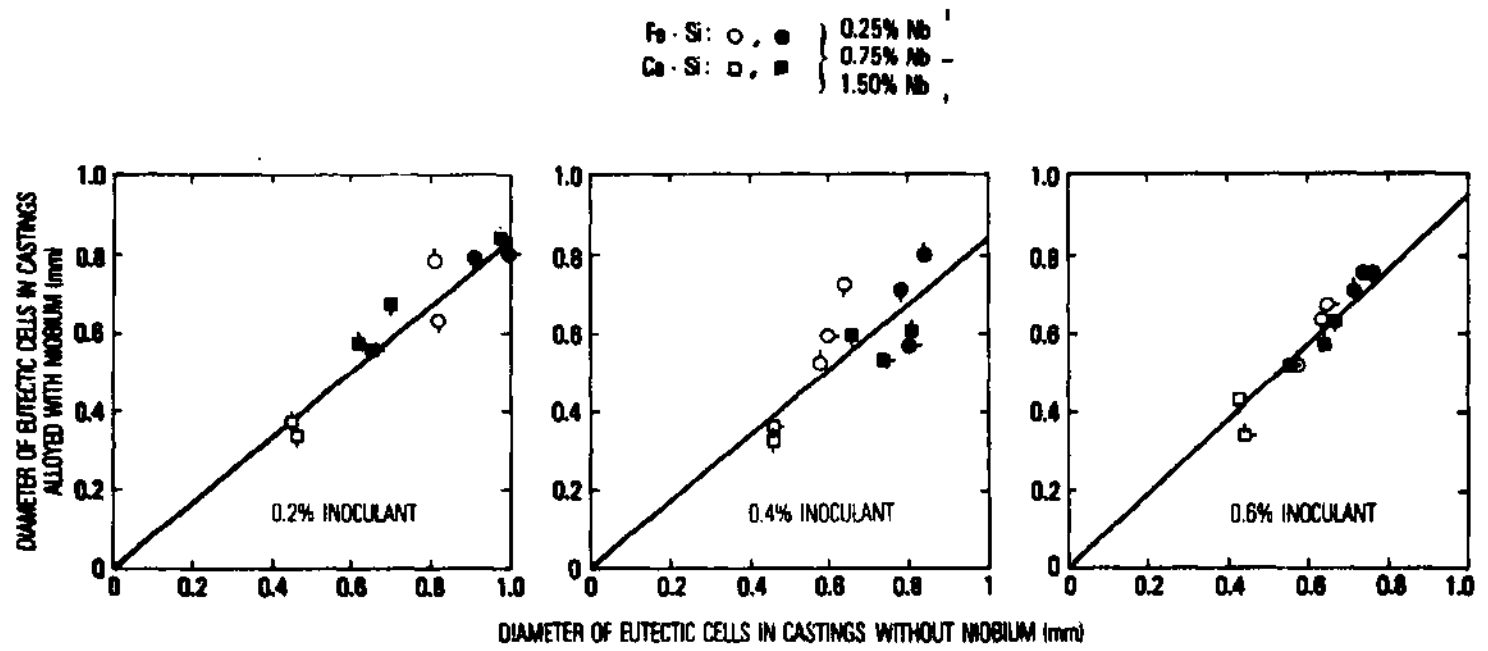


Fig. 5 — Diameter of eutectic cells in castings alloyed with niobium as a function of the cell sizes observed in control castings made without niobium.

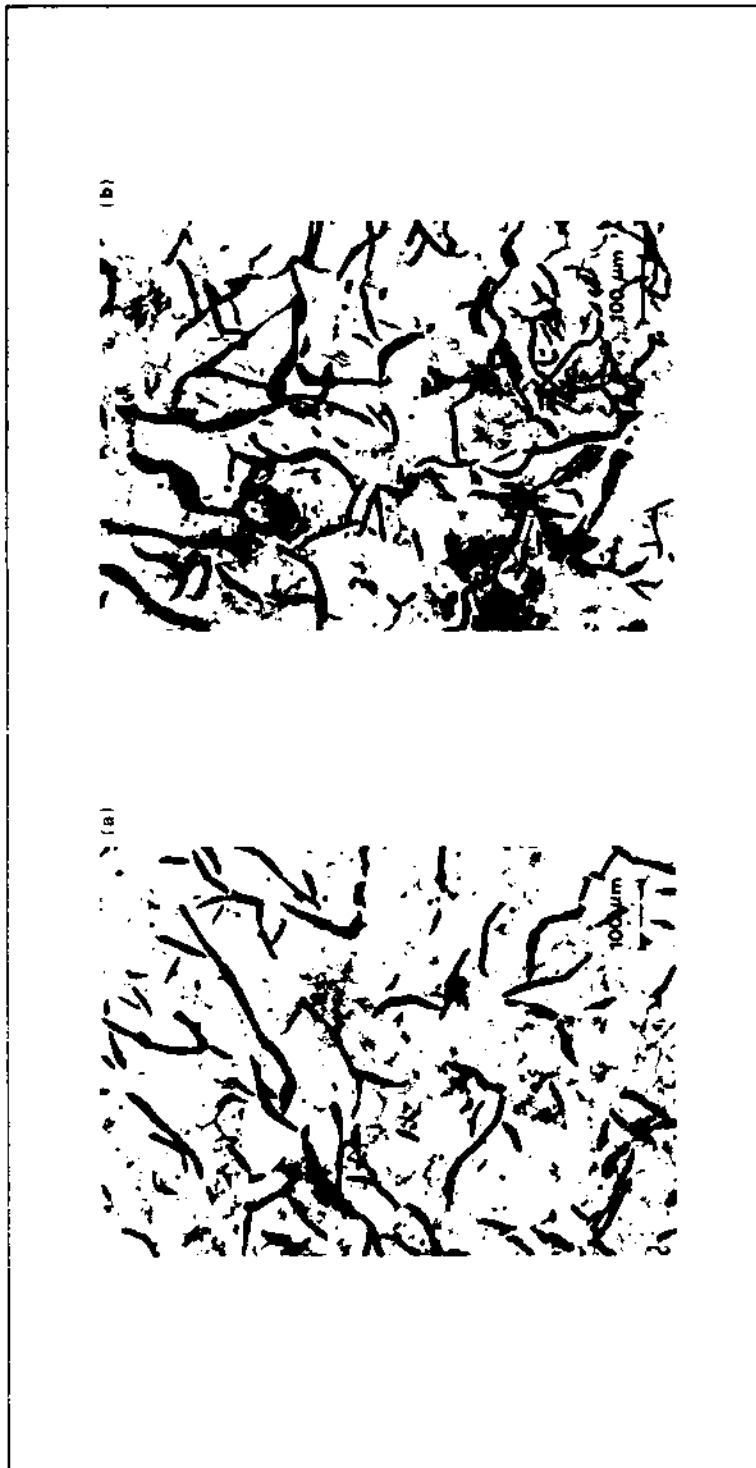


Fig. 6 — Optical photomicrographs Nb (C, N) carbides in casting alloyed with 0.8% Nb and inoculated with (a) 0.4% Ca-Si, or (b) 0.4% Fe-Si. Notice the carbides inside the austenitic dendrites.

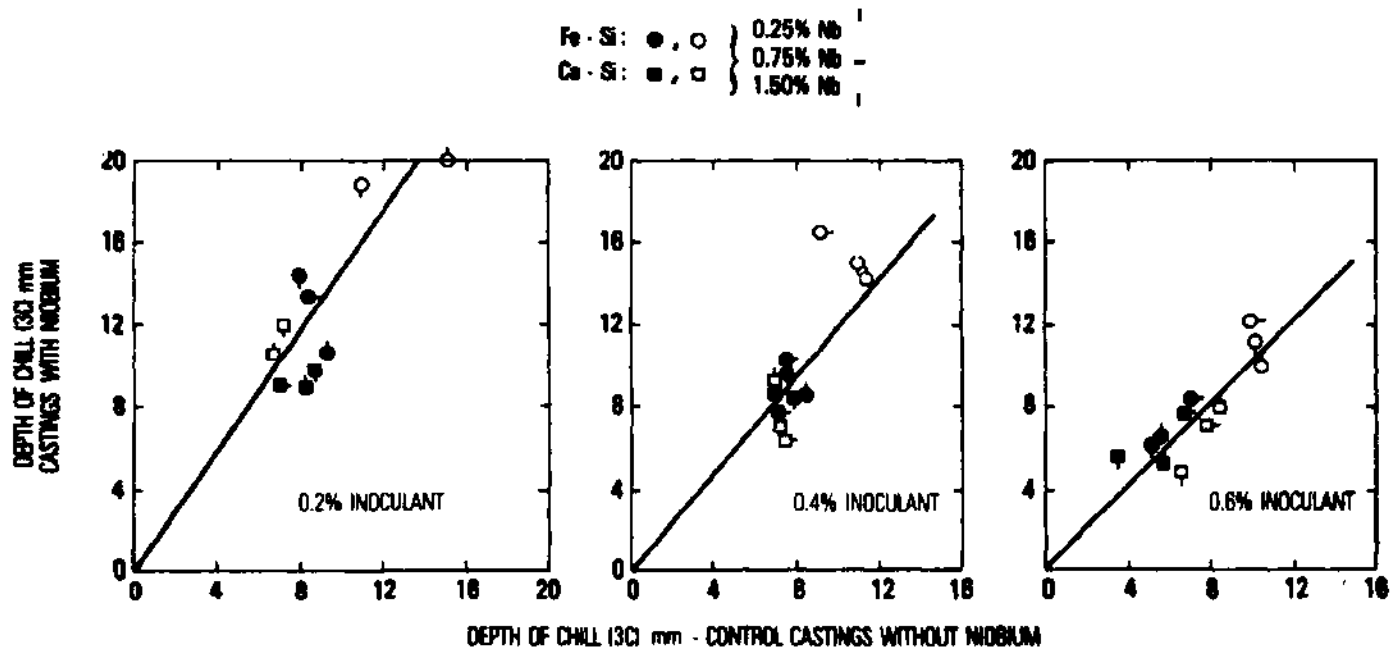


Fig. 7 — Chill depth (3-c) of castings alloyed with niobium as a function of that in control castings made without niobium.



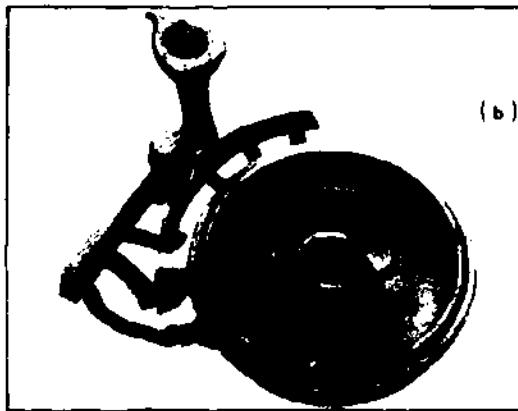
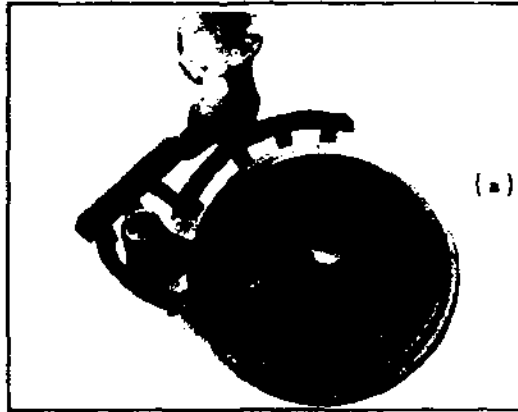
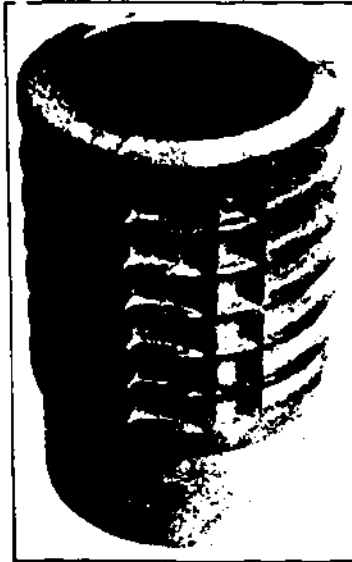


Fig. 8 — Brake drums cast with and without risers. 4.2% CE melt containing 0.47% Nb.



**Fig. 9 — Cylinder sleeve for Honda-125 motorcycle cast with a niobium alloyed melt.**

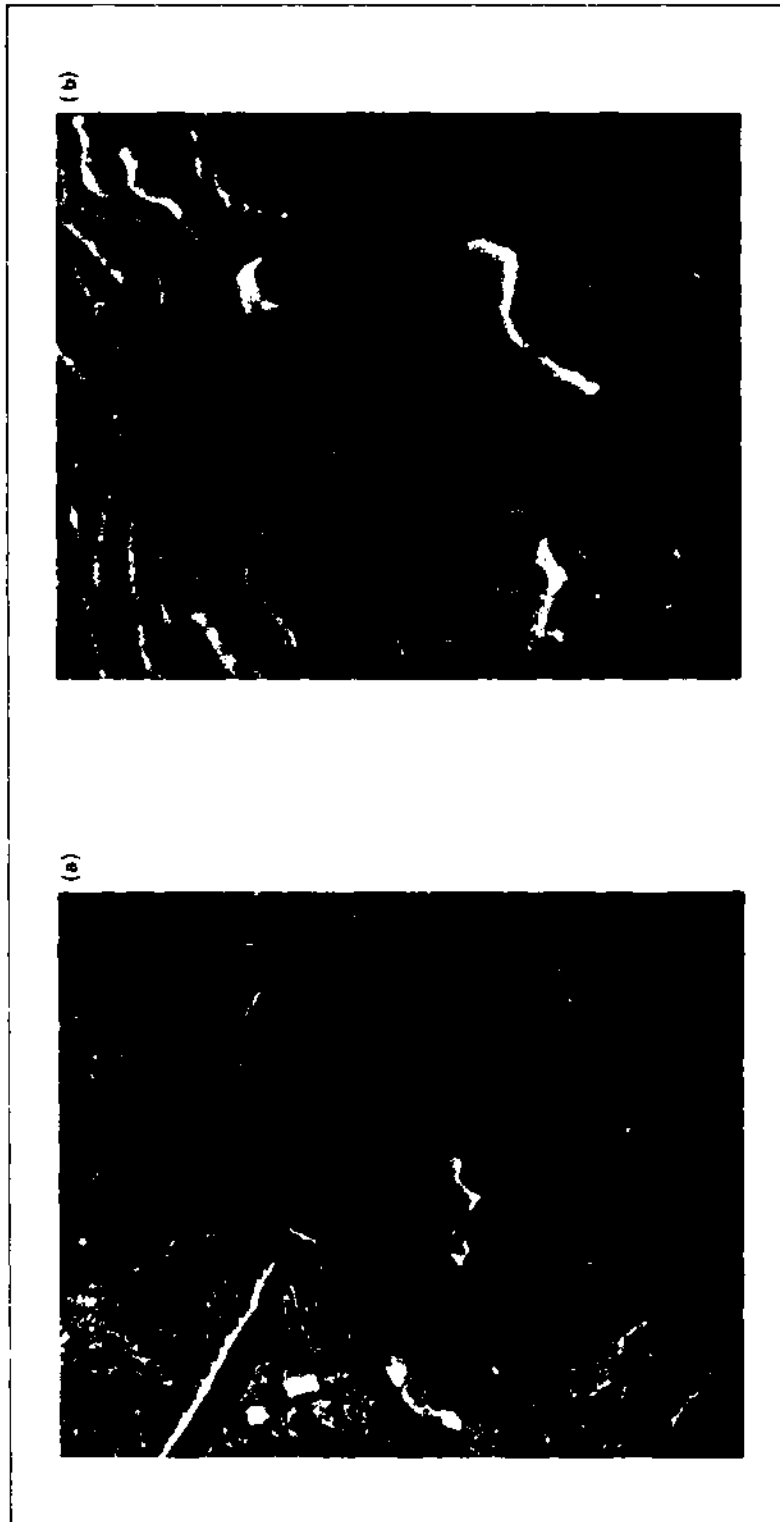


Fig. 10 — Scanning electron micrographs of the cylinder sleeve material. Details of (a) are shown in (b). NbC (1), sulfide (2) and phosphide (3).