

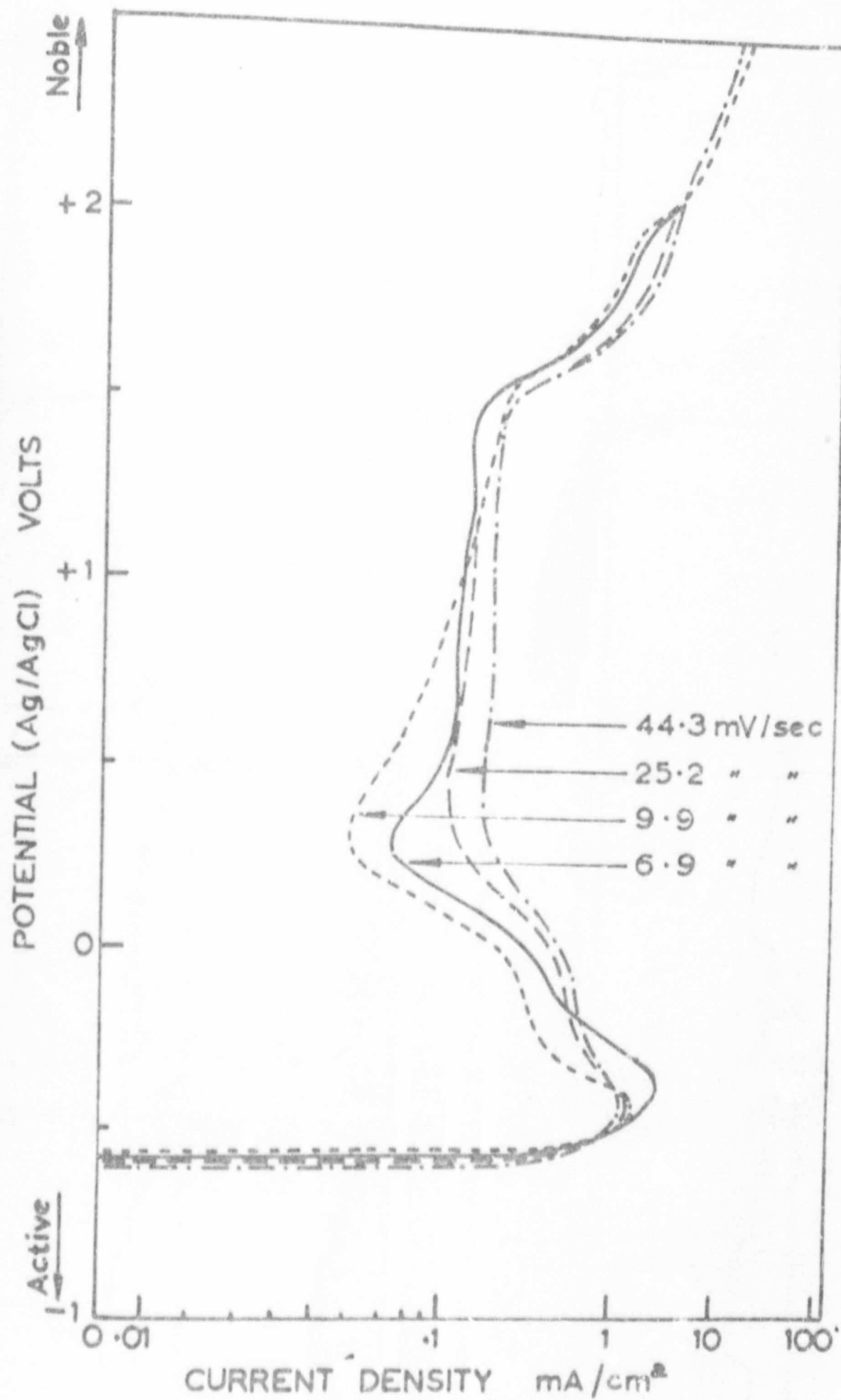


FIGURE 15: The influence of the scanning rate on the shape of the polarization curve for 15% ferrosilicon in 2.5% HCl.

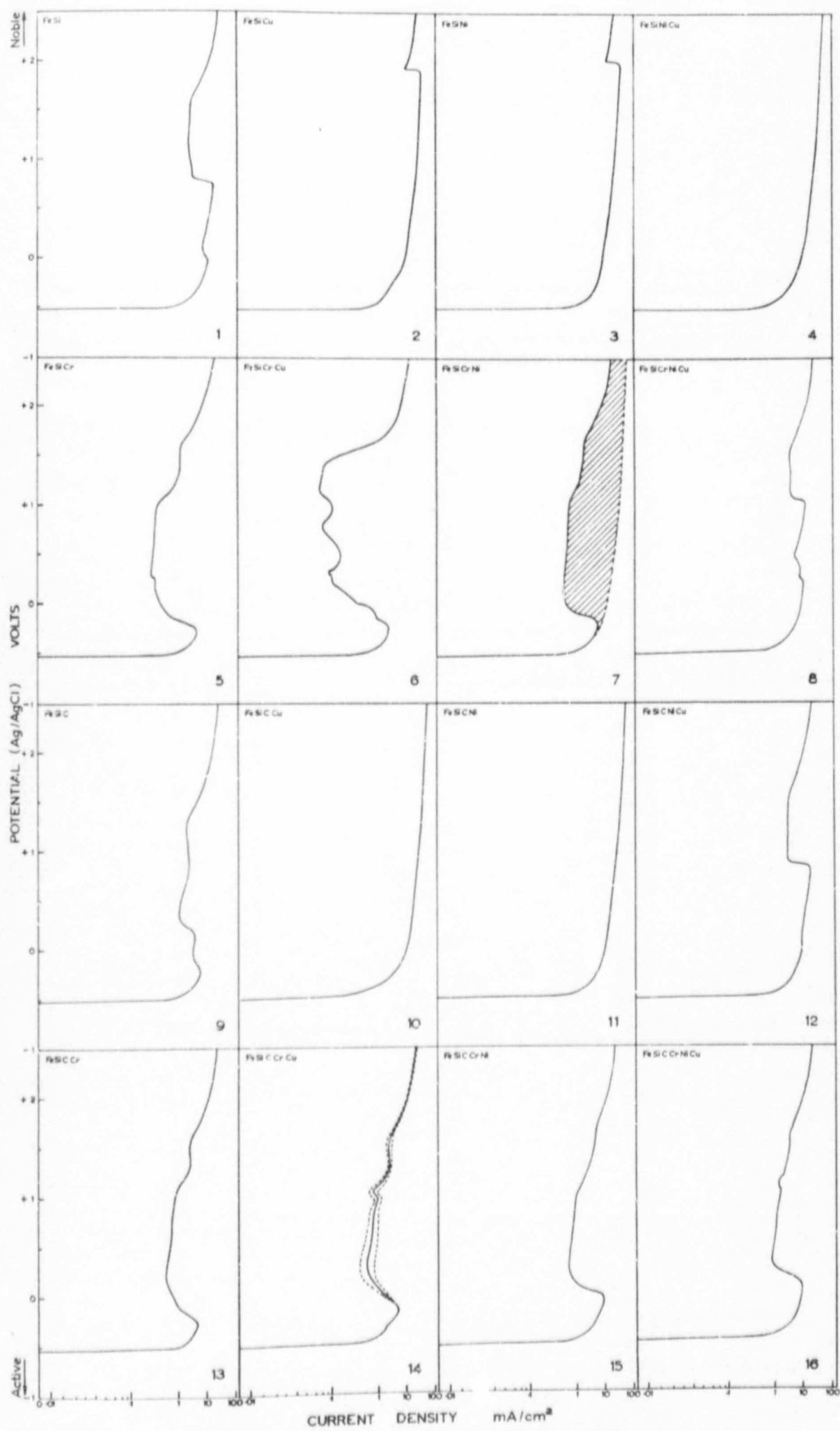


FIGURE 16: The polarization curves for specimens 1 to 16 in 2.5% HCl.

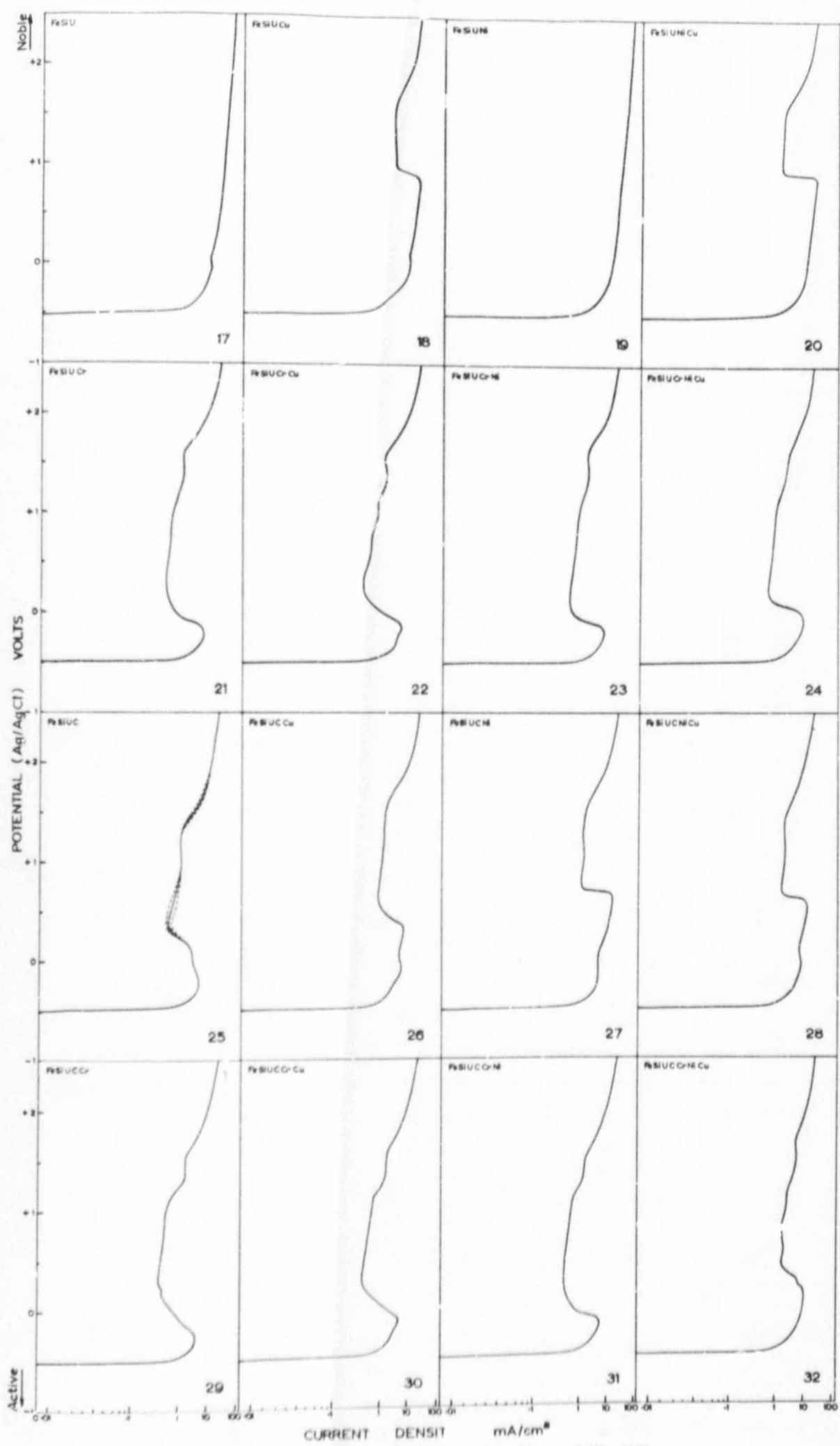


FIGURE 17: The polarization curves for specimens 17 to 32 in 2.5% HCl.

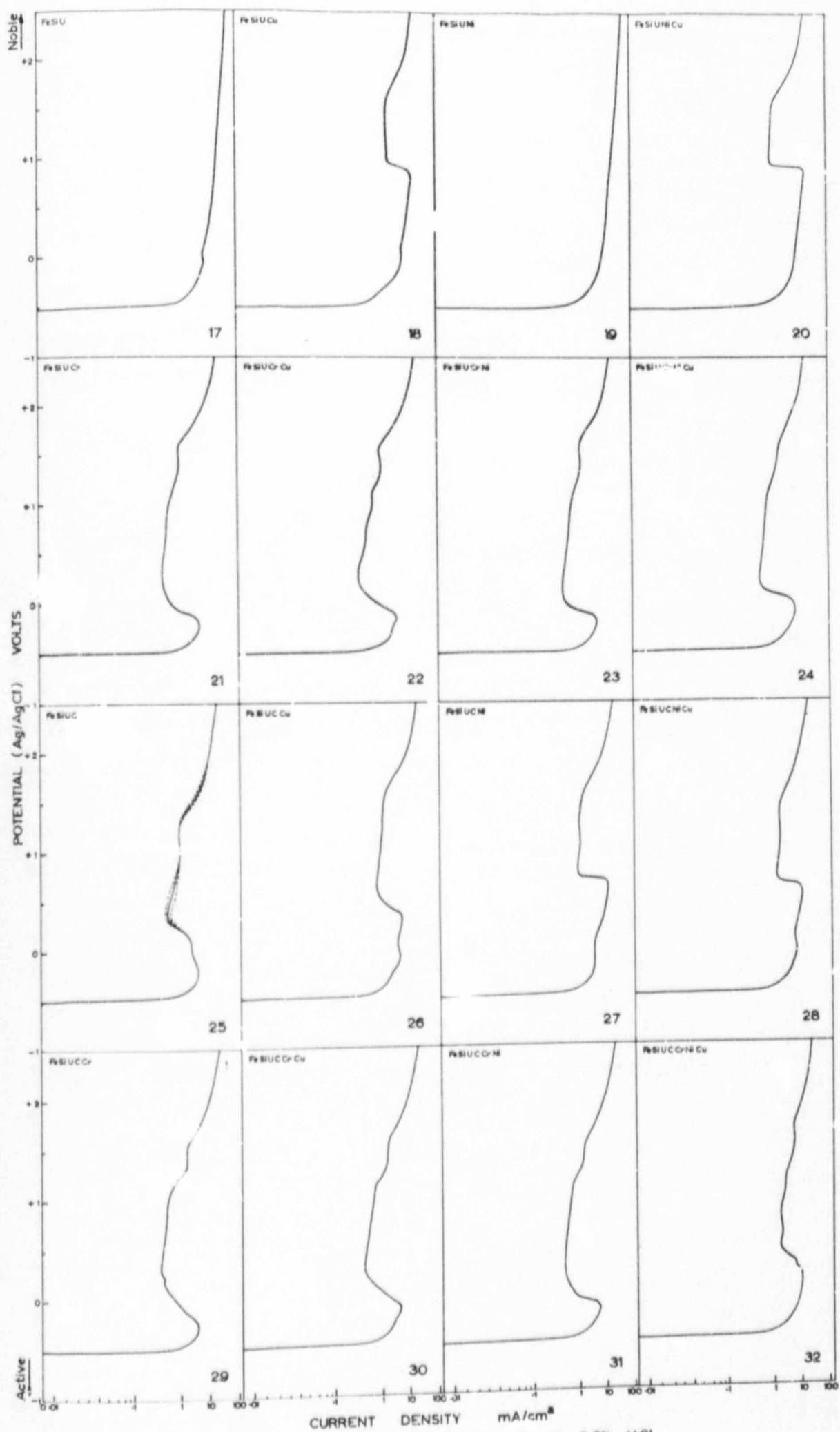


FIGURE 17: The polarization curves for specimens 17 to 32 in 2.5% HCl.

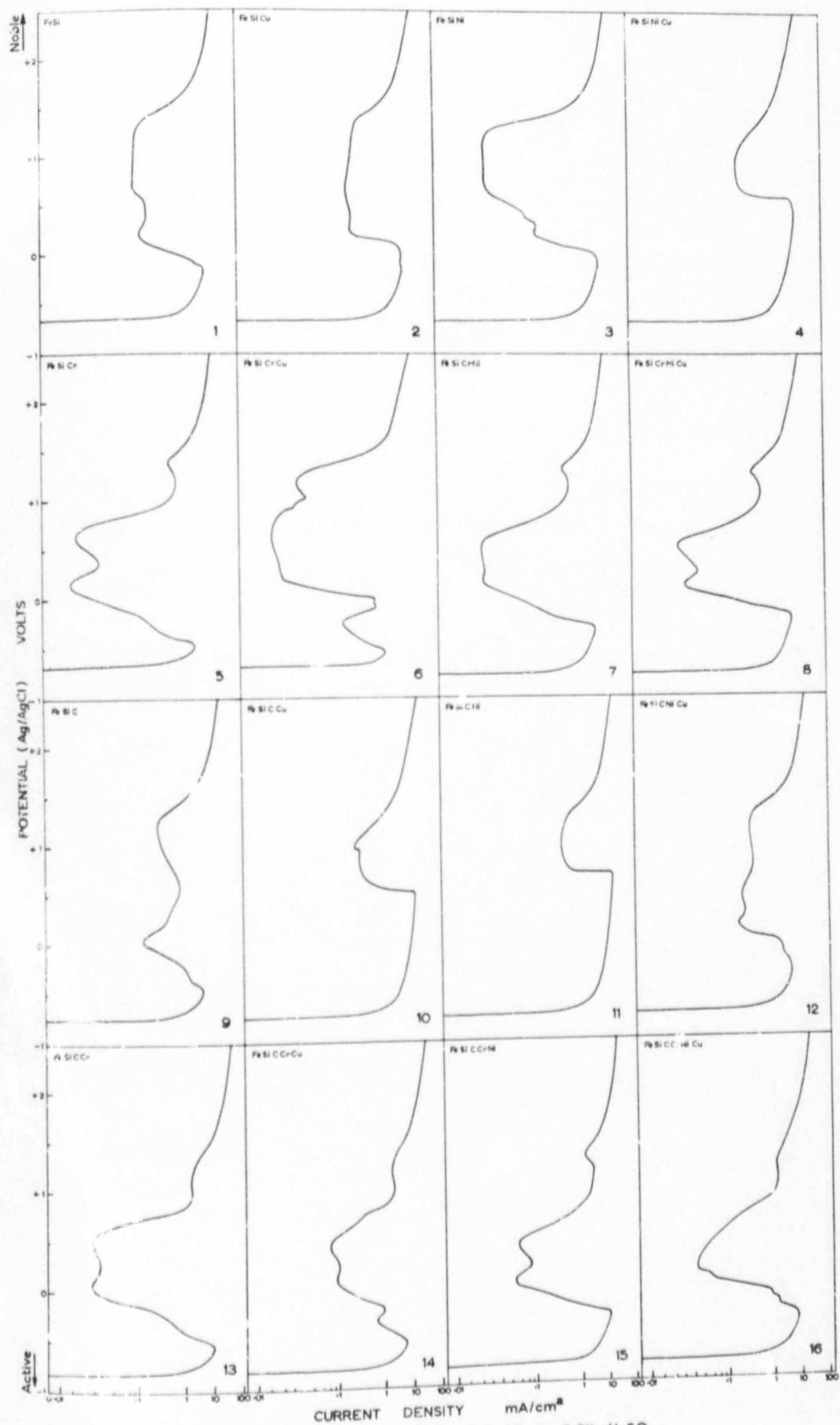


FIGURE 13: The polarization curves for specimens 1 to 16 in 2.5% H_2SO_4 .

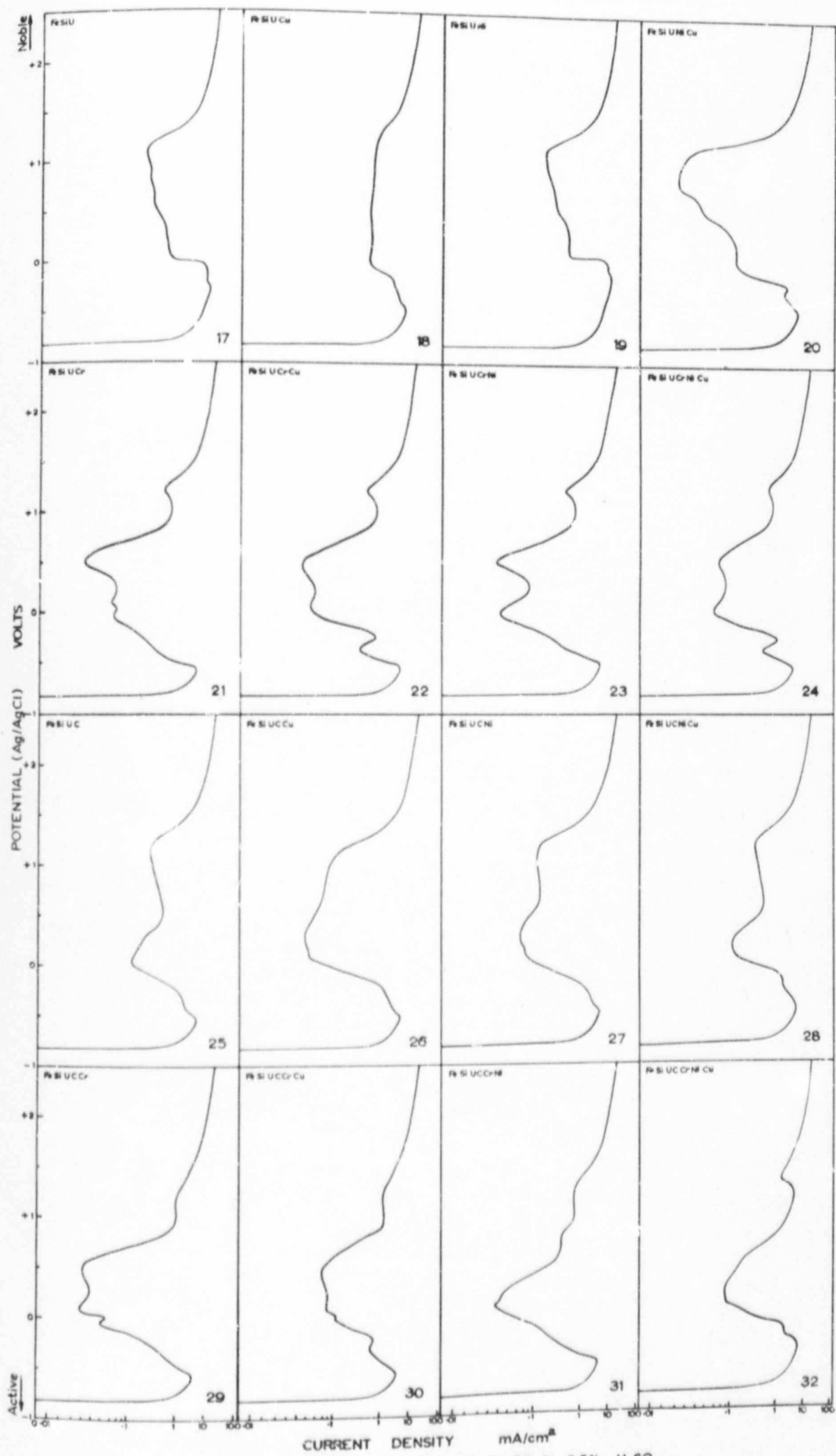


FIGURE 19 : The polarization curves for specimens 17 to 32 in 2.5% H_2SO_4 .



FIGURE 20: Polarization diagrammes showing influence of alloying elements on shape of curve. 12.5% Si alloys. 2.5% HCl.

and the specimen considered, was taken to be a measure of the passivation tendency - see Figure 16, specimen 7. The areas swept by the curves are given in Table 7.

The curves for heat treated specimens in HCl solution were not greatly different from the curves for the specimens as cast.

The influence of the silicon content on the shape of the polarization curve can be seen in Figure 21. These specimens were produced from steel containing approximately 0.1%C.

2.3.5 Discussion

The polarization curves were found to be characterized by good reproducibility. Specimen 25 in Figure 17 represents an average case while specimen 14 in Figure 16 shows poor curve reproducibility.

There are clearly large differences in the passivation tendencies of the different alloys as reflected by the curves of Figures 16, 17, 18 and 19.

The values are so consistent and agreement with the general picture of the corrosion resistance values of these alloys, so good, that the use of this method is considered justified. The areas to the left of the curves could equally well have been used as an indication of expected corrosion rate.

The use of these curves for semi-quantitative evaluations, has not been encountered in the literature.

TABLE 7 - Area by which the Polarization curve is removed from the Reference Curve of Specimen 4 in 2.5% HCl.

Specimen Condition Electrolyte		As Cast 2.5% HCl	Heat Treated 2.5% HCl	As Cast 2.5% H ₂ SO ₄
Specimen No.	Constituents	Arbitrary Units of Area (Proportional to Passivation Tendency)		
0	15% Si	39.0	-	-
1	12.5% Si	18.4	14.4	56.1
2	+ Cu	3.8	2.8	45.3
3	+ Ni	2.6	0	69.4
4	+ CuNi	0	0	29.4
5	+ Cr	47.1	50.7	81.0
6	+ CrCu	68.1	61.3	94.7
7	+ CrNi	39.2	33.8	70.8
8	+ CrCuNi	14.7	13.0	60.6
9	+ C	25.0	25.7	50.7
10	+ CCu	0	0	25.2
11	+ CNi	0	0	24.0
12	+ CCuNi	16.3	7.9	45.2
13	+ CCr	36.3	35.6	73.0
14	+ CCrCu	35.5	34.8	51.8
15	+ CCrNi	31.1	26.9	52.2
16	+ CCrCuNi	28.1	22.0	53.4
17	+ U	.5	1.0	43.6
18	+ UCu	17.9	12.4	41.4
19	+ UNi	0	0	42.1
20	+ UCuNi	19.0	13.0	75.3
21	+ UCr	41.8	36.8	69.3
22	+ UCrCu	41.5	37.5	63.3
23	+ UCrNi	38.1	39.9	66.0
24	+ UCrCuNi	33.9	29.0	57.5
25	+ UC	32.9	13.2	50.1
26	+ UCCu	30.7	13.4	67.0
27	+ UCNi	23.2	11.9	61.6
28	+ UCCuNi	22.3	14.7	46.2
29	+ UCCr	41.9	30.1	66.5
30	+ UCCrCu	39.4	34.5	54.2
31	+ UCCrNi	37.9	34.4	57.9
32	+ UCCrCuNi	22.3	13.2	41.6
33	+ 2% Mo	0	-	-
34	+ 4% Mo	0	-	-
35	+ C 2% Mo	0	-	-
36	+ C 4% Mo	0	-	-
37	+ Cr 2% Mo	32.2	-	-
38	+ Cr 4% Mo	5.9	-	-
39	+ C Cr 2% Mo	0	-	-
40	+ C Cr 4% Mo	5.8	-	-

The passivation tendencies of the different specimens can be seen in Figures 16-20. To compare the relative passivation tendencies of different specimens it is more convenient to study the figures of Table 7. On such an inspection it becomes evident that all alloys containing chromium show a strong passivating tendency - see large passivation figures for specimens 5-8, 13-16, 21-24 and 29-32. This beneficial influence of chromium on the corrosion resistance is further highlighted by comparing the alloys containing chromium (mentioned above) with their counterparts which do not contain chromium viz. specimens 1-4, 9-12, 17-20 and 25-28.

A comparison of the molybdenum alloys (23-40) with their counterparts not containing molybdenum (1, 9, 5 and 13) shows that this element is a deleterious addition. The addition of uranium to alloys containing carbon but not chromium, gave improved corrosion resistance - compare specimens 9-12 with 25-28. In the absence of chromium, the addition of copper and/or nickel results in decreased corrosion resistance - compare specimens 2, 3 and 4 with 1.

For all the alloys tested the passivating tendency is much greater in H_2SO_4 than in HCl.

The curves of Figure 21 clearly show how the passivating tendency of ferrosilicon depends on the silicon content. This tendency progressively increases as the silicon content is increased 7.5 to 15%. The 7.5% silicon alloy clearly shows no passivating tendency at all in 2.5% HCl, with the 15% silicon alloy showing a hundredfold decrease in current density in the passive region. The figures in Table 7 show that specimens 5, 6, 21, 22, 29 and 30 are no less corrosion resistant than the 15% silicon alloy.

2.4 TOTAL IMMERSION OF POWDER

2.4.1 Introduction

Long term weight change tests were carried out on powdered specimens to obtain somewhat more practical corrosion figures, even though plant conditions have not been duplicated.

2.4.2 Experimental Procedure

The specimens listed in Table 8 were ground and screened to -100 +150 Standard Tyler mesh. Triplicate 5 gram samples of each specimen were immersed in 50 milliliters of electrolyte containing 100 p.p.m. Cl as NaCl and 50 p.p.m. SO_4 as Na_2SO_4 . The glass

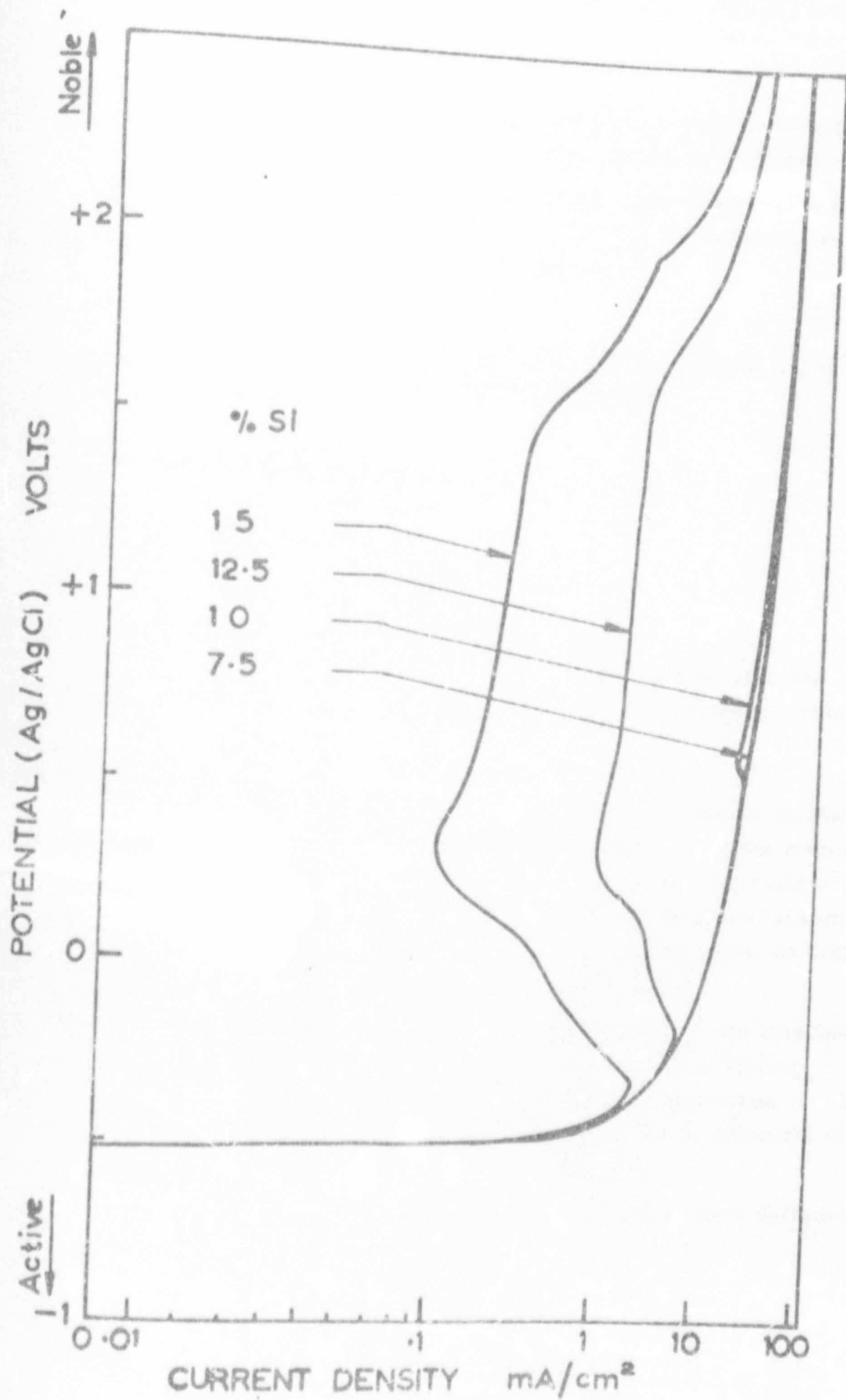


FIGURE 21: The influence of silicon content on the polarization curves in 2.5% HCl.

containers were shaken by hand once per day. The electrolyte volume was maintained constant by weekly additions of distilled water.

After 140 days immersion, the powder were treated with NaOH and the resulting precipitates separated from the remaining metal by magnetic means, for subsequent analysis.

2.4.3 Results

The specimens were inspected visually and arranged according to the apparent extent of corrosion as follows :-

- I No Visible Corrosion
- II Very Little Corrosion
- III Little Corrosion
- IV Medium Corrosion
- V Fairly Severe Corrosion
- VI Very Severe Corrosion

These symbols are presented in Table 8 together with the colorimetrically determined iron contents of the corrosion products.

2.4.4 Discussion

With the elements other than iron forming a relatively small part of each alloy, the iron content determination of the corrosion products is considered a sufficient indication of the relative corrosion rates, even if allowance is made for selective dissolution of the different elements. The loss of iron as given in Table 8, is therefore used for the comparison of the alloys.

Although there is a considerable difference in the triplicate figures A, B and C in Table 8, the test nevertheless clearly differentiated between specimens of different composition. The visual evaluations of the corroded specimens are in agreement with the relative values of the weight loss figures.

Heat treatment does not appear to have had a large influence on the corrosion rate.

TABLE 8 - Iron contained in the Corrosion Products of 5g Ground Samples of 12.5% Silicon Alloys after 140 days in an Aqueous Electrolyte containing 100 p.p.m. Cl and 50 p.p.m. SO_4 .

SPECIMEN			Fe in solution, mg.						Visual Rating
No.	Constituents	State	A	B	C	Ave.	On total sample	On Fe only	
1	Si		84.7	103.4	92.1	93.4	1.9	2.3	IV
4	Si/Ni/Cu		-	249.9	291.2	270.6	5.4	6.5	VI
6	Si/Cr/Cu		5.5	5.7	7.9	6.4	0.13	0.16	II
8	Si/Cr/Ni/Cu		91.2	82.1	104.8	92.7	1.9	2.3	IV
11	Si/Ni/C	AS	103.5	84.3	65.0	83.3	1.7	2.0	IV
14	Si/C/Cr/Cu	CAST	55.7	67.2	53.3	58.7	1.2	1.4	IV
18	Si/U/Cu		138.9	137.3	149.4	141.9	2.8	3.4	IV
21	Si/U/Cr		48.5	36.7	47.0	44.1	0.88	1.1	IV
22	Si/U/Cr/Cu		2.6	4.2	14.7	7.1	0.14	0.17	III
24	Si/U/Cr/Ni/Cu		10.8	12.0	12.9	11.9	0.24	0.28	III
28	Si/U/C/Ni/Cu		228.5	175.5	219.7	207.9	4.2	5.0	V
31	Si/U/C/Cr/Ni		57.0	58.6	56.8	57.5	1.2	1.4	III
1	Si		81.8	89.0	83.7	84.8	1.7	2.0	III
4	Si/Ni/Cu		201.1	258.6	254.5	238.1	4.8	5.8	V
6	Si/Cr/Cu		1.4	4.3	4.4	3.3	0.07	0.08	I
8	Si/Cr/Ni/Cu		107.3	107.1	102.7	105.7	2.1	2.5	IV
11	Si/Ni/C	HEAT	112.0	99.5	110.2	107.2	2.1	2.5	IV
14	Si/C/Cr/Cu	TREATED	52.8	99.0	85.3	79.0	1.6	1.9	IV
18	Si/U/Cu		151.9	134.2	153.3	146.5	2.9	3.5	IV
21	Si/U/Cr		48.0	45.9	41.9	45.3	0.9	1.1	IV
22	Si/U/Cr/Cu		13.4	9.2	8.1	10.2	0.20	0.24	II
24	Si/U/Cr/Ni/Cu		36.3	53.7	54.8	48.3	0.9	1.0	III
28	Si/U/C/Ni/Cu		172.7	280.0	262.5	238.4	4.8	5.8	V
31	Si/U/C/Cr/Ni		58.7	51.9	53.3	54.6	1.1	1.3	III

According to the figures as well as the visual rating symbols of Table 8, all the more corrosion resistant specimens contained chromium - specimens 6, 14, 21, 22, 24 and 31. The corrosion resistance of the 12.5% silicon alloy was not improved by additions of Ni, Cu, C and U in the absence of Cr, but these alloying elements in fact resulted in increased corrosion - compare specimens 4, 11, 18 and 28 with the unalloyed specimen 1 (Table 8).

CHAPTER 3

DISCUSSION AND CORRELATION OF THE RESULTS OF THE DIFFERENT TYPES OF TEST EMPLOYED

3.1 INTRODUCTION

The results of all the different corrosion tests are presented graphically in Figure 22. From these tables it is possible to see how the corrosion resistance of an existing alloy system is influenced by the introduction of a new element, in any one of three different electrolytes. In block 1 are given all the test values for specimens 1 to 32. Blocks 2 to 6 do not contain the values for the heat treated specimens.

In the first two columns of row 1, the corrosion properties of specimen 2 which contains 1% copper, are compared with those of specimen 1 which does not contain copper but is otherwise similar in composition. The passivation tendency decreases whereas the intercept current and powder weight loss figures increase on the addition of copper. An increased corrosion rate is thus clearly indicated. Similarly, the influence of copper on the Fe Si Ni alloy can be found by comparing the values for specimen 4 with those for specimen 3. Again the addition of copper results in an increase of corrosion. In the case of the Fe Si Cr alloy, the addition of copper proved to be beneficial - specimen 6 versus 5.

In the same way as block 1 summarizes the influence of 1% copper on various alloys, so is the effect of 1% nickel presented in block 2, that of 2% chromium in block 3, that of 0.5% carbon in block 4, that of 0.5% uranium in block 5 and that of 2 and 4% molybdenum in block 6.

Blocks 3 and 6 contain possibly the most interesting information. Block 3 illustrates the marked beneficial effect of chromium additions. Molybdenum, on the other hand, has a detrimental effect when added to 12.5% silicon alloys as is clearly shown in block 6. This is of particular importance since this finding is contrary to what would be expected in view of the excellent corrosion resistance of the 14.5% silicon alloy containing 3% molybdenum.

Blocks 1 and 2 show that both Cu and Ni are deleterious additions in the absence of chromium. From blocks 4 and 5 it appears that C and U may be mutually beneficial although the combined effect is still small compared to that of Cr.

A further example is given below to illustrate how values are read from Figure 22.

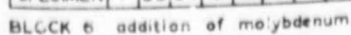
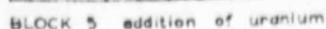
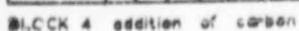
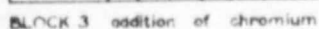
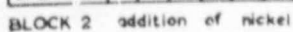
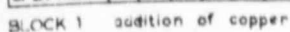


FIGURE 22: Graphic presentation of the influence of various alloying additions.

Instruction : Find the influence of nickel on an alloy containing Fe, Si, Cr and Cu in the amounts given in Table 3.

Procedure : The influence of nickel addition is given in the second block from the top in Figure 22. Specimen 6 is the Fe Si Cr Cu alloy and specimen 8 is the Fe Si Cr Cu Ni alloy. They appear adjacent to one another for ease of comparison. The presence of nickel can be seen to result in a decrease in the passivation tendency in HCl as well as in H_2SO_4 solutions, and an increase in the intercept current and powder weight loss in a $NaCl/Na_2SO_4$ solution.

Nickel is therefore shown to be a deleterious addition as regards corrosion resistance for the particular alloy considered.

3.2 COMPARISON OF THE TEST METHOD

The total immersion test is a traditional and direct method. It has the disadvantage of rather poor reproducibility, as shown in Table 8 but on the other hand it is strongly recommended because of the ease of interpretation of the results. It has provided a control for the somewhat abstruse results obtained electrochemically. Total immersion has the further disadvantage that observation of the change of weight with time is often not feasible. An unsuccessful attempt was made, with the aid of specific gravity bottles, to determine the dependence of corrosion rate on time.

Very satisfactory time studies were carried out using the over-voltage intercept method. Since these tests frequently covered a few days, they gave information on the ageing characteristics of the specimens, which is of particular importance in the case of ferrosilicon alloys.

The various electrochemical tests may either supplement or replace each other. Thus, the overvoltage intercept method was used for a dilute $NaCl/Na_2SO_4$ solution. A "full" polarization curve could not be drawn for this electrolyte due to excessive resistance polarization. On the other hand the "full" polarization curve was ideally suited to the HCl and H_2SO_4 electrolytes, and gave reproducibility of a high degree.

3.3 THE EFFECT OF ELECTROLYTE COMPOSITION

3.3.1 $NaCl/Na_2SO_4$ Solutions

Powder immersion tests were done on some of the specimens only. In eight cases it is possible to find from tests done by this method, the influence of the addition of one element. Referring to Figure 22, these are :-

Specimens 21/22 in top block,
6/8 and 22/24 in second block,
4/8 and 18/22 in third block,
6/14 in fourth block,
6/22 and 8/24 in fifth block.

Considering the overvoltage intercept test figures for the same specimen pairs, it is noticed that in six cases the two types of tests are in agreement whilst in the cases of specimens 21/22 and 6/22 they do not agree. The incidence of agreement is considered to be fair. The use of the overvoltage intercept method of corrosion rate determination as used here, seems justifiable as a screening test in the development of new alloys.

3.3.2 H_2SO_4 and HCl solutions

Ferrosilicon (or high silicon iron) is reported to have better resistance to attack by H_2SO_4 than to HCl. This more violent attack by HCl is ascribed to the readiness with which the relatively small Cl ions can perpetrate film breakdown.^{5,7} The "full" polarization curves of Figures 16, 17, 18 and 19 graphically demonstrate the extent of passivation of ferrosilicon alloys in these two acids. Table 7 summarizes the passivating tendencies of all the specimens and shows clearly that the passivating tendency is much greater for H_2SO_4 than for HCl. The same effect is evident in Figure 22.

3.3.3 A comparison of Polarization Test Results

Different polarization tests were performed in different electrolytes with each method best suited to the particular electrolyte. A comparison of the results obtained by these tests shows that the attack on the alloys by different electrolytes was apparently resisted equally well by the same alloying additions.

With reference to Table 21, it is found that :-

The overvoltage intercept	81%
tests in $NaCl/Na_2SO_4$	agreement
compared with the	in the direction
HCl "full" polarization	of current shift.
tests	

The overvoltage intercept tests in NaCl/Na ₂ SO ₄ compared with the H ₂ SO ₄ "full" polarization tests	give	75% agreement in the direction of current shift.
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The HCl and H ₂ SO ₄ "full" polarization tests compared	give	77% agreement in the direction of current shift.
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These figures show that generally speaking an alloying addition which results in an improvement of corrosion properties in one of the electrolytes considered, will in most cases be beneficial also for the other electrolytes.

3.4 EFFECTS OF ELEMENTS PRESENT IN THE ALLOY

3.4.1 Silicon and Uranium

Silicon is still the most important alloying element in the series of alloys investigated, as far as corrosion resistance is concerned. Both the overvoltage intercept method, and the "full" polarization method confirmed this well known beneficial effect.

The influence of uranium was erratic to such an extent that no conclusions can be drawn on the basis of the little information available. The highly reactive nature of the element could in some cases have an effect due to a degassing action, or the formation of stable carbides, thus partially or completely preventing the formation of graphite flakes.

3.4.2 Effect of cathodic and passivating additions

Thomashov^{14,15} has pointed out that cathodic constituents may either increase or decrease²⁸ the corrosion rate. A prerequisite for a decrease, is the possibility of anodic passivation which will occur only in the absence of high concentration of Cl⁻ ions.¹⁹

Both copper and nickel have been reported to be generally noble (i.e. cathodic) relative to 15% ferrosilicon.³ Considering these two elements as cathodic additions, and regarding the fact that in many cases in this investigation, they increased the corrosion rate, it appears that they assisted in accelerated anodic dissolution rather than passivation. This effect was however overcome by additions of chromium to the ferrosilicon alloy.

The general impression gained from Figure 21 is that the addition of chromium is always beneficial. This is not unexpected if it is borne in mind that chromium like silicon assists film build up, but to an even greater extent. Several alloys which were found to be particularly vulnerable to attack by solutions containing chloride ions, were greatly improved by the addition of chromium. These are alloy pairs 2/6, 3/7, 4/8, 10/14, 11/15, 17/21, 19/23.

3.4.3 The influence of molybdenum

As has been stated in 1.3, 3% molybdenum is reported to impart to 14.5% ferrosilicon good resistance to corrosion by HCl.

A reverse effect was found to occur on adding either 2 or 4% molybdenum to 12.5% ferrosilicon. Table 6 shows that specimens 33 to 40, which contain molybdenum, show no decrease in corrosion current with time after 1000 minutes immersion in 2.5% HCl, with the exception of specimen 37 where the drop in current with time is only very slight. The "full" polarization curves in Figure 20 (see also Table 7), indicate passivation tendencies in agreement with the overvoltage intercept values of Table 6. Specimen 37 is seen again to exhibit the greatest passivation tendency. Figure 22 graphically illustrates the deleterious effect of molybdenum on 12.5% ferrosilicon.

The following explanation is advanced for this apparent discrepancy :-

Molybdenum which stabilizes at a potential even more noble than that of copper in a NaCl solution,¹⁴ may very well act as a cathodic addition by establishing on the surface of the alloy, sites which are noble relative to the ferrosilicon background. The arguments given in 4.2 for the action of a noble addition such as copper, would then also apply to molybdenum even though its position in the electromotive force series is far more active than that of copper.

The addition of a cathodic constituent to a non-passivating system, will increase the corrosion rate. As can be seen from Figures 1 and 21, a 12.5% ferrosilicon alloy has a relatively low passivating tendency and could thus react accordingly. On the other hand, the addition of 14.5% silicon results in an alloy of greatly improved passivating tendency and could react favourably to the introduction of a cathodic addition. Molybdenum appears

to act as such by further improving the corrosion resistance of this alloy.

3.4.4 The influence of carbon

Carbon has been found by some workers^{2,7} to be a corrosion promoting component of ferrosilicon. With a few exceptions, this was found to be the case also with the alloys tested in this investigation, notwithstanding the fact that graphite is a cathodic element and could under certain circumstances, as already described in the previous sections, cause a decrease of corrosion rate.

The required conditions are almost certainly not met. Referring to the graphite filled cavities mentioned in 1.3 it is practically certain that the electrolyte within the cavities will contain insufficient oxygen for complete passivation of the metal. The cathodic element, graphite, will have a destructive effect on the metal rather than preserving it by stimulating the formation of a protective layer. The corrosion will be further accelerated by the formation of internal differential aeration cells in the graphite cavities.

3.5 THE EFFECT OF HEAT TREATMENT

Considering the information in Tables 3, 4 and 5 it is seen that heat treatment resulted only in relatively small changes in corrosion resistance. It is conceivable that flame spheroidizing of finely crushed particles, which results in complete or partial melting of the particle, could have a much more drastic effect on the crystal structure and phase changes, than resulted from the relatively mild treatment to which the specimens were subjected.

3.6 ALLOYS DEVELOPED

Referring to Tables 6, 7 and 8, and Figure 22, the alloys with the best corrosion resistance appear to be 5, 6, 21, 22 and 31, all of which contain chromium. As indicated by the overvoltage intercept and "full" polarization methods, the corrosion resistances of these alloys are comparable with that of 15% ferrosilicon. The specific gravities of these alloys are at the same time somewhat higher than the 7.00 value for the 15% silicon alloy. Thus specimen 6 has a specific gravity of 7.20 and specimen 31 has a value of 7.17. This increase is significant because it is reflected in the specific gravity of the heavy medium suspension which will be increased from 4.0 (say) to about 4.1 without an increase in the viscosity of the medium.

3.7 PRACTICAL EVIDENCE OF THE IMPORTANCE OF CORROSION RESISTANCE

The importance of corrosion resistance of the ferrosilicon powder is underlined by the following example :-

A commercially available heavy medium powder which is used extensively and which was recommended especially for its high specific gravity, gave the figures in Table 9.

TABLE 9 - Analysis and Specific Gravity of new and used Heavy Medium Powder

ELEMENT	NEW	USED
Si	13.3%	12.7%
Cu	3.36%	2.80%
Ni	3.36%	2.41%
Fe	78.4%	76.6%
TOTAL	98.42%	94.51%
S.G.	7.19	6.45

The used material was recovered by the magnetic separator in the plant. It is evident from the figures in the table that a considerable amount of corrosion took place, with the corrosion product still adhering partly to the particles, thus lowering the specific gravity considerably.

It is interesting to note that the composition of this alloy is rather similar to that of specimen 4 in the series of this investigation, viz. 12.5% Si, 1% Cu, 1% Ni. Specimen 4 exhibited very poor corrosion resistance.

CHAPTER 4

CONCLUSIONS

It has been established that a substitute for 15% ferrosilicon can be made with a comparable corrosion resistance and somewhat higher specific gravity. This was achieved by alloying a 12.5% silicon alloy with small amounts of certain elements, notably chromium.

The addition of molybdenum to the low (12.5%) silicon series of alloys was found to be deleterious. Copper, nickel, uranium and carbon may either improve or worsen the corrosion resistance depending on the other elements present.

The passivating tendencies of the ferrosilicon alloys decreased as the corrosivity of the medium was increased. This was found by testing in a neutral solution such as $\text{NaCl}/\text{Na}_2\text{SO}_4$, and then in acids such as 2.5% H_2SO_4 and 2.5% HCl . The increased corrosion in the acid media was undoubtedly due to the reduction of the SiO_2 rich film by the nascent hydrogen produced during the corrosion process.

The agreement of the polarization test results with conventional weight loss figures, justifies the use of these highly reproducible tests for the investigation of the properties of different ferrosilicon alloys.

By using the anodic polarization curves, a complete test can be done in about a quarter of an hour, giving a semi-quantitative value for the passivation tendency. If it is important that the rate of passivation be known, it can be determined by means of the overvoltage intercept method. About one day is sufficient for this, with a longer period being required for finding the ultimate extent of passivation. Such ageing tests were carried out on all the specimens and the results were found to be in agreement with the published literature.

The electrochemical tests proved to be reliable accelerated qualitative tests. The anodic polarization curves were used to evaluate passivation tendencies, whereas the actual rate of passivation was determined by the overvoltage intercept method.

The influence of alloy composition on the magnetizing and demagnetizing characteristics was not determined.

Author Du Plessis David Johannes

Name of thesis The Polarization And Corrosion Characteristics Of Ferrosilicon Alloys Used As A Heavy Medium.

PUBLISHER:

University of the Witwatersrand, Johannesburg

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