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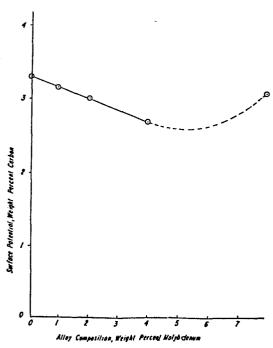
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- 54 Iron-nickel-chromium-molybdenum alloy.
- (57) A highly carburization resistant alloy characterized by good structural stability at elevated temperatures, containing about 24% to 35% nickel, about 19 to 25% chromium, about 1.5 to 6% molybdenum, carbon in an amount not exceeding about 0.12%, up to 1.5 or 2% manganese, up to 1% aluminium, up to 1% titanium, up to 1% silicon and up to about 0.3% nitrogen, the balance, apart from residual amounts of deoxidizing and cleaning elements, and impurities, being iron.





Iron-nickel-chromium-molybdenum alloy.

The subject invention is directed to a novel iron-nickel-chromium (Fe-Ni-Cr) alloy characterized by a high degree of resistance to carburization and which affords a combination of other desirable metallurgical properties, including structural stability at elevated temperatures of ca. 980-1095°C, the ability to be both hot and cold worked, good resistance to corrosion including resistance to chloride attacks, etc.

INVENTION BACKGROUND

As is known, iron-base, nickel-chromium alloys are extensively

10 used in a host of diverse applications by reason of one or more (and
within limits) strength, ductility, corrosion resistance, etc. Such
attributes notwithstanding, this type of alloy generally suffers from an
inability to resist satisfactorily the destructive toll occasioned by
carburization, a phenomenon by which the alloy structure is

15 environmentally degraded from the surface inward. As a consequence, the
load bearing capacity of the alloy is adversely affected as manifested by
impaired strength (stress rupture, creep), lowered ductility, etc.
Usually the initial attack is along the grain boundaries and this tends
to accelerate failure, or at least premature removal of a given alloy

20 component from its operational environment.

In any case, if the carburization problem could be substantially minimized without subverting other properties, such an alloy would find expanded use for such applications as the petrochemical and coal gasification fields, ethylene pyrolysis, etc., areas in which alloys are exposed to a combination of carbonaceous environments and high temperature.

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But in addressing the problem of carburization resistance, it would be self-defeating to achieve success at the expense of other desired properties as contemplated herein, e.g., high temperature structural stability over prolonged periods of time, elevated temperature stress-rupture strength, workability, etc.

SUMMARY OF THE INVENTION

It has now been discovered that an iron-nickel-chromium alloy of special chemistry and containing carefully correlated percentages of iron, nickel, chromium, molybdenum and carbon and certain other constituents discussed herein results in a (i) markedly enhanced carburization resistant material at temperature levels at least as high as 980-1095°C. Moreover, the subject alloy is (ii) workable, (iii) not prone to form deleterious amounts of topological closepacked phases prematurely such as sigma, and otherwise offers (iv) structural stability over substantial periods of time upon exposure to elevated temperature. Further, the alloy is (v) weldable and (vi) affords a high degree of resistance to pitting attack in aggressive corrosive media.

In addition to the foregoing, it has been also found that the

contemplated alloy offers enhanced oxidation resistance, a phenomenon by
which the alloy surface undergoes attack in oxygen-containing
environments at high temperature. As a consequence, the material
continuously undergoes weight loss, the surface "spalls off." As would
be expected the oxidation problem is particularly acute in "thin section"

mill product forms, strip, sheet, thin wall tubing, etc.

DESCRIPTION OF THE INVENTION

Generally speaking, the subject invention contemplates an iron-nickel-chromium alloy containing about 24% to 35% nickel, about 19 to 25% chromium, about 1.5 to 6% molybdenum, carbon in an amount not exceeding about 0.12%, up to 1.5 or 2% manganese, up to 1% aluminum, up to 1% titanium, up to 1% silicon and up to about 0.3% nitrogen, the balance, apart from residual amounts of deoxidizing and cleaning elements, and impurities, being iron.

Unless otherwise indicated, all percentages in this specification and claims are by weight.

In carrying the invention into practice, molybdenum plays a major positive role in maximizing resistance to carburization. Advantageously, the molybdenum content should be maintained at a level of about 2% or more in seeking optimum carburization resistance. Percentages much beyond 4% do not offer an appreciable advantage in this respect, given cost considerations, and generally it will not exceed 4.5%. However, where resistance to corrosion, particularly to chloride attack, is important, the molybdenum can be as high as about 6%.

Chromium imparts resistance to corrosion but should not exceed about 24 or 25% since it lends to sigma formation at elevated tempera20 ture and attendant embrittlement problems. A range of 20-23% is quite satisfactory. The total chromium plus molybdenum content preferably does not exceed 26% or 27% since molybdenum also lends to sigma formation. Where high temperature applications are not involved, the chromium plus molybdenum content can be extended to 29%.

Nickel contributes to good workability and mechanical properties. Should the nickel level fall much below 24% the stability of the alloy could be impaired, particularly if the chromium and/or molybdenum is at the high end of their respective ranges. On the other hand, nickel percentages above 35% (up to 42%) increase cost without significant property degradation. A nickel range of 28% to 35% is considered most beneficial.

Carbon in excess, say 0.3%, detracts from pitting resistance. In addition, workability is adversely affected; however, carbon does add to strength and other properties and, accordingly, a range of about 0.04 or 0.05 to 0.1% is deemed distinctly advantageous.

For workability and other benefits titanium should be present, but amounts above 1% are not required. A range from 0.1 or 0.2 to 0.75% is quite beneficial. Aluminum can be used as a deoxidizer and as an aid to workability. A range of 0.05 to 0.5% is quite satisfactory.

By so controlling the carbon, titanium, and aluminum as well as the high percentage constituents (Mo, Cr, Ni) the alloys are not only workable but can be produced using air melting practice. This is not to say vacuum processing is precluded but there is an economic advantage in the former.

Manganese and silicon can both be present in amounts up to 2% and 1%, respectively. Higher amounts are unnecessary. Where oxidation resistance is of importance manganese should not exceed about 0.6%. Manganese promotes weldability, particularly at the higher end of its range with aluminum at the lower end of its range. It is deemed that nitrogen, a potent austenite former, can be present, a range of 0.05 to 0.25% being considered satisfactory. Nitrogen is considered to be beneficial at the lower nickel levels.

One advantageous composition comprises about 28 to 35% nickel, 20 to 24% chromium, at least 1.5% and up to 4.5% molybdenum, carbon 25 present up to 0.12%, titanium present up to 1%, up to 1% aluminum, up to 2% manganese, up to 1% silicon and up to 0.3% nitrogen, the balance, apart from impurities and residual deoxidizing and cleaning elements, being iron.

Impurities that may be present are those usually associated with alloys of this type, in amounts that do not adversely affect their basic characteristics.

The following information and data are given as illustrative of the invention.

Carburization Resistance

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14kg. samples of various compositions were air melted and forged, the compositions being given in Table 1, Alloys A, B and C being beyond and Alloys 1 and 2 being within the invention.

TABLE I

Alloy	Мо	С	Cr	N1_	Ti	Al	Mn	Si
A	0.01.	.06	21.01	31.84	.38	.30	.14	.23
В	0.92	.06	20.96	32.16	.37	.32	.11	.18
1	1.98	.12	20,27	32.27	.35	.26	.26	.27
2	3.94	.14	19.93	32.49	.31	.25	.37	.32
C	7.87	.11	20.32	32.45	.34	.31	.30	.46

Balance iron plus impurities, e.g., sulfur and phosphorus In respect of the above alloy compositions, they were subjected to a gaseous carburization test in which specimens were machined into cylinders approximately 12 mm diam. and 25 mm in length. These were placed in a tray and put into a muffle type furnace, the temperature being 982°C. The test was conducted for 100 hours using a gaseous atmosphere of 2% methane plus hydrogen. After exposure, the samples were water quenched and then weighed to determine weight gain data. The results are reported in Table II.

TABLE II

Carburization Data: Normalized Weight Gain

		Мо	Weight Gain
25	Alloy	(%)	(mg/cm ²)
	<u> </u>	.01	11.7
	B .	0.92	9.3
	1	1.98	6.3
	2	3.94	6.2
30	С	7.87	4.9

As can be observed from the data in Table II, a rather dramatic improvement obtained in respect of carburization resistance with regard to Alloys 1 and 2. Alloy C (7.87% Mo) showed some further improvement but the cost associated with such molybdenum levels would not likely warrant such percentages on a commercial scale.

Weight gain is essentially a measure of how many atoms of carbon have been absorbed but without regard as to to the depth of effect.

Thus, concentration versus depth profiles were determined and Figure 1 reflects this information. Figure 1 confirms, in essence, the data of Table II. As is manifest, with increasing molybdenum percentages the penetration profile shrinks indicating that less diffusion has occurred.

Figure 2 depicts surface potential versus molybdenum content. This may be viewed as the chemical effect of molybdenum on carbon diffusion, or specifically the effect of molybdenum on gas-metal reaction at the surface, carbon solubility, or carbon activity coefficient. The surface potential appears to be a quite linear decreasing function of molybdenum, at least up to 4%. The behavior at 8% molybdenum is not clearly understood.

We have also determined that molybdenum decreases the carbon diffusion coefficient.

Oxidation Resistance

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Tables III (chemistry) and IV (data) afford a comparison of the oxidation resistance behavior of alloys within the invention versus commercial (control) alloys of somewhat similar composition.

The oxidation test was one of cyclic oxidation using 14 kg.

samples (air melted) forged to flats, hot rolled to 7.9 mm and cold
rolled to 3.2 mm. The test comprised subjecting specimens for 15
minutes at 1093°C, cooling for 5 minutes in air, heating again to 1093°C
holding for 15 minutes, again cooling 5 minutes in air, until testing was
completed. Specimens were checked at 100 hr. intervals. Prior to test
the specimens were annealed at 1177°C and water quenched. Oxide was
removed by grinding to 120 grit.

Alloy	Mo Z	C %	Cr Z	N1 Z	Ti Z	A1 Z	Mn Z	Fe %
3	1.89	.05	20.82	32.73	.30	.32	.09	Bal.
4	3.92	.04		32.37		.29	.08	**
D	9.62	.04	20.70	32.40	.35	.28	.08	**
Control	#1	.05	21	32	.5	.5	1.0	71
	#2* .01		20.93	_			.10	11

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*Contained .54 Si and .07 Cu

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TABLE IV

1093°C Cyclic Oxidation Data

15	Heat No Alloy						a, mg/cm ² r. 700h		Depth of Ohr. Attack (mm)
	3	+1.0	+1.4	+2.0	+2.3	+1.7	-24.9	-81.7	0.10
	4	+1.1	+1.6	+2.2	+2.6	+3.1	-15.5	-66.9	0.15
	D	-0.3	-0.4	-0.2	-0.2	-0.8	-8.9	-40.2	0.13
	Control								
20	#1	+2.6	-40.1	-86.	6 - 124	.4 -156.8	-223.1	-316.4	0.51
	Control								
	#2	+1.5	- 1.7	-25.	0 -65	.3 - 98.8	-180.9	-294.5	0.48

As will be observed, the alloys within the invention compared more than favorably with the Control alloys. Maintaining manganese at low levels, i.e., below 0.6 or 0.5% contributes to enhanced oxidation resistance.

Cyclic oxidation Test on Alloy 4 in the form of 0.76 nm thick sheet also compared favorably with Control Alloy No. 1 as reflected in Table V.

30		TABLE V											
	Alloy				Data, 0 400hr.			1000hr.					
35	4 *Control	+1.6	-0.1	-21.1	-26.4	42.5	-75.5	-95.3					
	#1	+2.6	-40.1	-86.6	-124.4 -	-156.8	-223.1	-316.4					
	*= 3.18 mm	gauge											

Testing of thin gauge specimens is markedly more severe because warpage is much more likely to occur on cooling thus increasing the tendency for oxide scaling.

Structural (Phase) Stability

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In Table VII infra are given the results of various impact (ability to absorb impact) tests. Charpy V-Notch impact testing is often used as a means of predicting whether an alloy will undergo embrittlement on being exposed to elevated temperatures for prolonged periods.

While a 1000 hour test period might normally be deemed sufficiently severe, tests were also conducted for 3000 hours at temperatures of 760°C and 816°C. The composition of the alloys tested are given in Table III.

TABLE	VI
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	Alloy	Мо	С	Cr	Ni	T1	A1	Mn	<u>Fe</u>
15	3	1.89	.05	20.82	32.73	.31	.32	.09	Bal.
	4	3.92	.04	20.85	32.37	.40	.29	.08	Bal.
				20.70					

TABLE VII

	Temperature	Time	Charpy V	-Notch, k	kg/cm²		
20	(°C)	(hr.)	Alloy 3	Alloy 4	Alloy D		
	649	1000	19.7	13:5	3.1		
	760	1000	11.2	9.7	0.5		
	760	3000	15.5	2.4	*		
	816	1000	15.0	6.2	*		
25	816	3000	14.0	2.9	*		

*discontinued

All samples annealed at 1177°C and water quenched prior to exposure.

The alloys of the invention (Alloys 3 and 4) were quite resistant to premature embrittlement as evident from Table VII. Even upon 3000 hour

testing the alloys within the invention performed satisfactorily. Alloy D (9.62% Mo) did not stand up at 760°C'/100 hr. It was sigma prone.

centrifugally cast hollow billet was extruded to a tube shell and cold worked to 5.7 mm dia. x 6.9 mm wall tube. (Composition: 0.06 C, 0.03 Mm, 0.33 Si, 31.98 Ni, 21.55 Cr, 0.18 Al, 0.32 Ti, 3.12 Mo, Fe balance). The specimen was annealed at 1177°C for an hour and air cooled prior to test. The tube was rupture tested at 649°C/8.5 kgf/mm² for the tremendously long period of 26,394 hours (3 years) and then discontinued, no failure having occurred. A metallographic study showed M₂₃C₆ carbides and very fine particles of sigma within the grains which were deemed innocuous, particularly since a portion of the specimen was placed in a vise and bent to ascertain if embrittlement had occurred. The ductile nature of the specimen was obvious.

15 Weldability

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Compositions for weldability are given in Table VIII. In this connection, two alloy series were evaluated one involving variations in aluminum and manganese (Alloys 5-8), the other (Alloys A, B, 1, 2, and C) exploring the effect of molybdenum.

Material was provided as ½" thick x 2" wide hot forged flats which were overhauled and rolled to 7.87mm thick x 50.8 mm wide for Varestraint test samples. Included for purposes of comparison is a well known commercial alloy (Control).

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TABLE VIII

	Alloy	Мо	С	Cr	Ni	Ti	A1	Mn	Fe
	A	0.01	.06	21.01	31.84	.38	.30	.14	Bal.
	В	0.92	.06	20.96	32.16	.37	.32	.11	Bal.
5	1	1.98	.12	20.27	32.27	.35	. 26	.26	11
	2	3.94	.14	19.93	32.49	.31	.25	.37	**
	C	7.87	.11	20.32	32.45	.34	.31	.30	71
	5	3.93	.05	20.32	32.14	.40	.27	.07	**
	6	3.82	.05	21.08	32.25	.31	.04	.15	11
10	7	3.90	.05	20.50	32.14	.42	.30	.56	**
	8	3.87	.08	20.88	32.25	.28	.04	.56	**
	Control Alloy	.26	.08	19.89	32.80	.44	.32	.83	tt

*contained 0.04% copper. All heats contained small amounts Si.

Bal. = balance and impurities.

A travel speed of 12.7 cm/min, an amperage of 190 amps and a voltage over the range of 13.8-15.0 volts were employed. The Varestraint test, one of relatively considerable severity, was conducted on both a 127 cm and 63.5 cm radius block with the results given in Table IX.

TABLE IX Varestraint Test Results

	12	7 cm.	Radius	Block		63	.5 cm.	Radiu	s Bloc	k
	Test		Av.		Av.	Test		Av.		Av.
Alloy	Thick	MCL	MCL	TCL	TCL	Thick	MCL	MCL	TCL	TCL
-	(mm)	(mm)	(mm)	(mm)	(mm)	 (mm)	(mm)	(mm)	(mm)	(mm)
	7.70	0		0		7.67	0.46		2.01	
A	7.72	0	0	0		7.70	0.33	0.38	1.27	1.90
	7.70	0	0	0		 7.72	0.38		2.44	
	7.72	0		0		7.98	0.30		0.66	
В	7.85	0	0	0	0	8.00	0.38	0.36	1.14	1.35
	7.82	0		0		 7.95	0.38		2.21	
		_		_						
_	7.98	0		0	_	7.98	0.76		2.67	0.40
1	7.90	0	0	0	0	7.92	0.51	0.61	0.81	2.13
	7.87	0		0		 7.90	0.56		3.15	
		_		_						
_	7.85	0	_	0	_	8.13	0.89		1.73	
2	7.98	0	0	0	0	8.13	0.63	0.79	2.36	2.18
	7.98	0		0		 8.00	0.84		3.00	
		•		•		7 00	0.04		4 00	
_	7.98	0	- 40	0		7.98	0.91	0.05	4.09	
С	8.00	0	0.18	0	0.33	7.95	0.71	0.86	2.46	3.12
	7.98	0.53		1.02		 7.95	0.96		2.84	
	7 05	0		0		7 05	0 20		0 51	
-	7.95	0	•	0	•	7.95	0.30	0 40	0.51	2 24
5	7.95	0	0	0	0	8.00	0.66	0.48	2.90	2.21
	7.95	0		0	·	 8.03	0.71		3.25	
	7.62	0		0		7.59	0.71		2.44	
c			Λ	0	0	7.67		0.20		2 07
6	7.62	0	0	0	U	7.07	0.66	0.38	3.12	2.97
	7.70	0				 				
	7.95	0		0		7.98	0.96		3.20	
7	8.00	0	0	0	0	7.95	0.56	0.76	1.65	2.44
	0.00					 1.55	0.30	0.70	1.05	
	7.72	0		0		7.77	0.41		1.60	
8	7.80	Ŏ	0	Ö		7.72	0	0.20	0	
J	7.75	Ö	Ū	Ö		_	_	0.00	_	
						 				
	7.77	1.19		2.57		7.70	0.96		5.0	
Control	7.77	0.66	.89	1.04	1.80	7.70	0.96		5.0	
Alloy	7.85	0.81		1.83		7.80	0.76		3.33	

MCL = Maximum Crack Length TCL = Total Crack Length

Amperage 190 Voltage 13.8 - 15.0

Travel Speed 12.7 cm/min.

All the specimens performed at least as (more) satisfactorily as the commercial control alloy. Of the molybdenum series, the high

molybdenum material (Alloy C, 7.87% Mo) was more susceptible to cracking. Regarding the Al/Mn series, the low aluminum, high manganese material (Alloy 8) was the most crack resistant. Accordingly, by using molybdenum levels within the invention, particularly with low aluminum, 0.04 to 0.35, and high manganese, say 0.3 to 0.6%, weldability is improved. Pitting Corrosion Resistance

Data reported in Table X give an indication of pitting resistance. Samples were cold-rolled to 3.46mm and annealed at either 1177°C or 1288°C for one hour, followed by water quenching. Specimens (approx. 17.75 x 7.6 mm) were prepared by grinding to 320 grit and then exposed 4 hours at 35°C in acidified 10.8 2/o FeCl. 6H₂ O (Smith Test). After exposure, weight loss per unit surface area was determined and the specimens visually evaluated for the appearance of pits.

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						TABLE	<u> </u>				
15	Alloy	C Z	Mo 7	Cr %	ni Z	Ti Z	Al X	Mn Z	Si Z	Pitting	m'g/cm²
	E	.29	1.98	20.86	32.70	.42	.33	.11	1.84	Yes	n.d.
	9	.05	1.89	20,82	32.73	.30	.32	.09	.20	Yes	n.d.
	F	.28	3.79	20.95	32,28	.29	.29	.07	.17	Yes	7.773
20	10	.04	3.29	20,85	32.37	.40	.29	.08	.21	No	0.334
	G	. 28	2.26	20.86	32.47	.32	.31	.07	.18	Yes	10.181
	n.d.	= not	deter	mined							

As can be seen from Table X, carbon at the higher levels is detrimental to pitting resistance. It detracts from the resistance to pitting imparted by molybdenum. Accordingly, where corrosion resistance is important carbon should not exceed about 0.12% Also, for such purposes the molybdenum can be extended to 6%.

Irrespective of carburization resistance and other attributes, if the alloys are unworkable, then they would find little utility. However, alloys within the invention are both hot and cold workable. Using Alloys 3, 4 and D of Table VI, these alloys forged readily and the forgings upon inspection were of high quality.

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Hardness data are given in Table XI for given annealing temperatures. Also included is hardness in the cold worked condition. In this connection, specimens were cold rolled to about 3.2 mm thick from thickness given in Table XII.

TABLE XI

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		Annealing Heat	Annealing Hardness	As Cold Worked	
	Alloy	Treatment,	Rb	Hardness, Rc	
	3	1177°C/1 hr.	66.5	33	
		1177°C/1 hr.	71.5	33	
10	D	1177°C/1 hr.	84.5	33	

TABLE XII

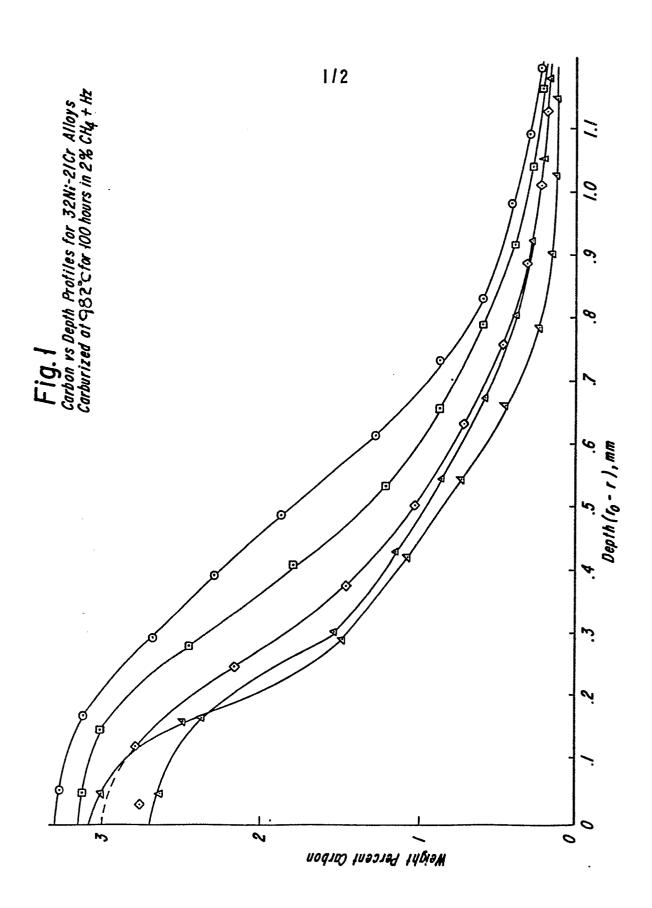
	Alloy	Thickness	(mm)	Thickness (mm)	ZReduction
	3	13.3		3.20	76
15	4	12.0		3.23	73
	D	12.7		3.18	75

Considering both the data from Tables XI and XII, the hardness measurements reflect that the alloys are relatively readily workable. From Table XII, it will be noted that cold reductions of more than 60% could be achieved without intermediate annealing. This together with the hardness data reflects that the alloys have excellent cold workability and a low work hardening rate. It might be added that high carbon is not beneficial to workability.

CLAIMS

- 1. An iron-nickel-chromium-molybdenum alloy consisting, by weight, of about 24% to 35% nickel, about 19 to 25% chromium, about 1.5 to 6% molybdenum, carbon in an amount not exceeding about 0.12%, up to 1.5 or 2% manganese, up to 1% aluminum, up to 1% titanium, up to 1% silicon and up to about 0.3% nitrogen, the balance, apart from residual amounts of deoxidizing and cleaning elements, and impurities, being iron.
- An alloy according to claim 1 in which the nickel content is from 28 to 35%, the chromium content is from 20 to 24%, the
 molybdenum content does not exceed 4.5%, and titanium is present.
 - 3. An alloy according to claim 2 containing 29 to 33% nickel, 20.5 to 23% chromium, about 2 to 4% molybdenum, 0.04 to 0.1% carbon and 0.2 to 0.75% titanium.
- 4. An alloy according to any preceding claim in which the sum of chromium plus molybdenum does not exceed 26%.
 - 5. An alloy according to any preceding claim in which the aluminum content does not exceed 0.5% and the manganese content is from 0.5 to 1%.
- 6. An alloy according to any preceding claim in which the 20 aluminum content is from 0.04 to 0.35% and the manganese content is from 0.3 to 0.6%.
 - 7. An alloy according to any preceding claim which contains nitrogen up to about 0.25%.
- 8. An alloy according to any preceding claim in wrought 25 form.

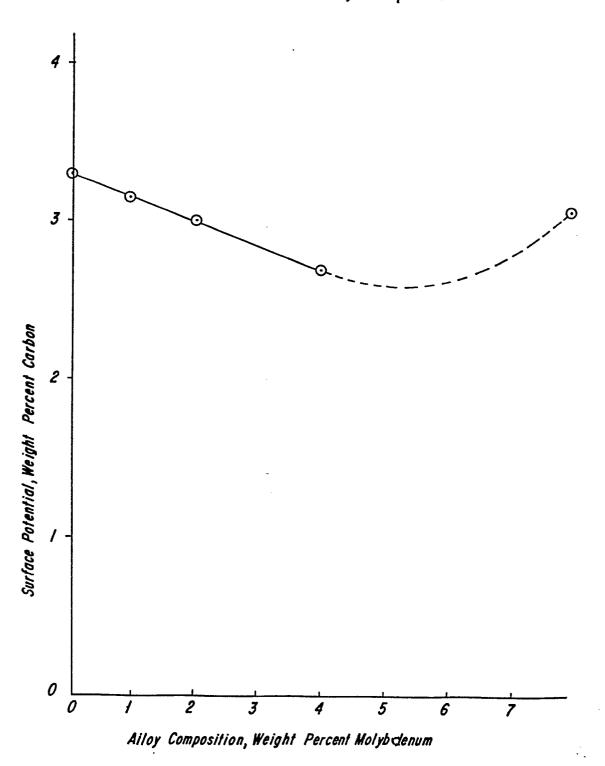
- 9. The use of an alloy according to any preceding claim for applications requiring a combination of resistance to carburisation and structural stability at elevated temperatures.
- 10. The use of an alloy according to claim 5 or claim 6 for 5 welded products requiring a combination of resistance to carburisation and structural stability at elevated temperatures.



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Fig. 2

Effect of Molybdenum Content on Surface Potential During Carbon Diffusion in 32N; 21Cr Alloys at 982°C



European Patent Office

EUROPEAN SEARCH REPORT

EP 85 11 4419

Category		h indication, where approp		Relevant	CLASSIFICATION OF THE
alegory	of relev	ant passages		to claim	APPLICATION (Int. Ci 4)
х	GB-A- 638 007 ELECTRIC INT. CO * Claims 1,4,7; column, pargraph page 9, lines 1-	.) page 8, left h under tab	-hand	1	C 22 C 38/44 C 22 C 30/00
x	* Page 4, table 1-6; alloys 3987			1,4	
x	GB-A- 741 558 EDELSTAHLWERKE A * Claims 1-4,6 *	Ġ)		1,2	
x	 GB-A- 531 466 * Claim 1 *	- (ETCHELLS)		1-3	
x	US-A-3 175 902 * Claims 1,2 *	- (FERREE)		1	TECHNICAL FIELDS SEARCHED (Int. CI 4) C 22 C
x	GB-A-2 117 792 * Claims 1,2 * 492, & DE - A - A - 4 489 040	& FR - A -	2 524	1,7	
x	GB-A- 993 613 JERNVERKS AB) * Claims 1,3,6,9	•		1,4,7	
		-,	/-		
	The present search report has t	peen drawn up for all claim	s		
Place of search THE HAGUE Date of completion of the search 13-03-1986				LIPPE	Examiner INS M.H.
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EUROPEAN SEARCH REPORT

0184038 Application number

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