

(19)



(11)

EP 2 935 639 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:

16.11.2016 Bulletin 2016/46

(51) Int Cl.:

C22C 38/02 (2006.01) **C21D 9/14** (2006.01)
C22C 38/44 (2006.01) **C22C 38/46** (2006.01)
E21B 17/02 (2006.01)

(21) Application number: **13811174.5**

(86) International application number:

PCT/EP2013/076740

(22) Date of filing: **16.12.2013**

(87) International publication number:

WO 2014/095747 (26.06.2014 Gazette 2014/26)

(54) **BAINITIC STEEL FOR ROCK DRILLING COMPONENT**

BAINITSTAHL FÜR GESTEINSBOHRKOMPONENTEN

ACIER BAINITIQUE POUR COMPOSANT DE FORAGE DE ROCHES

(84) Designated Contracting States:

**AL AT BE BG CH CY CZ DE DK EE ES FI FR GB
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO
PL PT RO RS SE SI SK SM TR**

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(43) Date of publication of application:
28.10.2015 Bulletin 2015/44

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Description

TECHNICAL FIELD

5 **[0001]** The present invention relates to a bainitic steel according to the preamble of claim 1. The present invention further relates to a drill rod component according to the preamble of claim 7. The present invention further relates to method for manufacture a drill rod component according to the preamble of claim 10. The present invention also relates to the use of the inventive bainitic steel according to the preamble of claim 15.

10 BACKGROUND ART

[0002] Drilling rods for mining and construction work typically comprises a central rod portion, a threaded male end and a threaded female end. In operation, a drilling head or drilling bit is screwed onto the male end of the rod and the drilling head is driven into the rock or ground by a drill rig. One type of drilling is the so called "top hammer drilling" in which the drilling rig is arranged to provide high rotational movement and percussion to the drill rod. As the length of the drill hole proceeds, the drill rod may be extended by screwing further drill rods onto the end of the precedent one.

[0003] Drill rods may be manufactured by forging and threading the ends of a steel rod into mating male and female connectors. However, the most common practice today is to manufacture the male and female connectors separately and then attach the connectors with friction welding to a respective end of a steel rod.

20 **[0004]** One problem related to drill rods is their relative short service life, since the rate by which the drill rods wear out and have to be replaced, has a direct impact on the total cost for the drilling operation. A further problem is the strength of the rod. If a rod breaks, it may take considerable time to retrieve it from the drill hole.

[0005] In the past some work has been done to improve drill rods. For example, WO97/27022 is directed to the problem of soft material zones occurring in the interface between the connector and the central rod after friction welding. When a connector and a central rod are friction welded together, heat evolves in the interface between connector and central rod. The heated zone is referred to as "the Heat Affected Zone", (HAZ). In the HAZ the steel material is annealed and a zone of soft material occurs in the interface between rod and connector. The soft zone becomes the weakest part of the drill rod and is typically the position where the drill rod breaks. To solve this problem, WO97/27022 proposes a steel in which the chemical composition has been balanced such that the hardness of the most tempered portion in the HAZ has a hardness equal to the core hardness of the drilling rod.

[0006] The steel described in WO97/27022 has lead to improvements in the service life of drill rods, in particular in view of failure in the interface between connector and central rod. However, the overall service life of drill rods is still not sufficient.

[0007] Field observation has shown that today failure in drill rods rarely occurs in the interface between connector and central rod. Instead, the life length of the drill rods seems to be limited by failure in the threaded portion of the connectors.

[0008] Consequently, it is an object of the present invention to solve at least one of the above problems. In particular it is an object of the present invention to achieve an improved steel composition which allows for the manufacturing of drill rods with long service life. A further object of the present invention is to achieve a cost effect drill component which can be used over a long period of time. It is also an object of the present invention to achieve a method for producing wear resistant drill components. Yet a further object of the present invention relates to the use of the improved steel composition in rock drilling components.

SUMMARY OF THE INVENTION

45 **[0009]** According to the invention at least one of these objects is met by a bainitic steel comprising (in weight%):

	C:	0.16-0.23
	Si:	0.8-1.0
	Mo:	0.67-0.9
50	Cr:	1.10-1.30
	V:	0.18-0.4
	Ni:	1.60-2.0
	Mn:	0.65-0.9
55	P:	≤ 0.020
	S:	≤ 0.02
	Cu:	< 0.20

(continued)

N: 0.005 - 0.012 wt%

balance Fe and unavoidable impurities.

[0010] The inventive steel is primarily intended for producing case hardened components that are subjected to repeated wear at elevated temperatures, i.e. 300 - 500°C, for example case hardened threaded connectors in drill rods. These components have a martensitic surface zone and a bainitic-martensitic core.

[0011] Results from field test performed during top hammer drilling have shown that case hardened drill rods manufactured from the inventive steel last surprisingly longer than drill rods manufactured from conventional steel.

[0012] During top hammer rock- or soil drilling above ground, the drill rod is subjected to intensive percussion from the drilling rig. The percussion causes a shock wave which progresses through the interconnected drill rods down to the drill bit in the bottom of the hole. As the shock wave progresses through the interconnected rods, approximately 5 % of its energy is lost in the form of heat that mainly evolves in the threads of the male and female connectors of the interconnected drill rods. Consequently, the working temperature in the connectors during top hammer drilling is high, typically up to 300°C but it may reach 500°C. In above-ground top hammer drilling, air is typically used for cooling the drill rods and also for removing the drill cuttings. However, air is not an effective cooling fluid and does not cool the rods sufficiently to avoid that the evolved heat causes the martensitic case in the threads of the connectors of the drill rods to transform into the softer phases cementite and ferrite. In conventional drill rods, the transformation of the martensite may cause the surface of the threads to soften and eventually cause the connectors to wear out. As adhesive wear resistance is in direct relation to the hardness.

[0013] The reason for the surprisingly long service life of the drill rods manufactured from the inventive steel is not entirely understood. However, without being bound by theory, it is believed that the balanced amounts of the alloy elements silicon, molybdenum, chromium and vanadium in the steel causes the martensitic surface of the drill rod connectors to retain the hardness at the high working temperatures during top hammer drilling.

[0014] Silicon stabilizes epsilon carbide and retards therefore the transformation of the hard martensitic surface zone of the connectors into softer cementite and ferrite up to temperatures of approximately 300°C. However, as the temperature rises in the connectors during drilling, the martensitic phase in the surface of the case hardened connectors will eventually start to transform into cementite and ferrite. The amount of martensite in the surface zone of the connectors therefore drops and consequently also the hardness of the surface zone drops. During the transformation of the martensite into cementite and ferrite, carbon is released into the steel.

[0015] In the inventive steel the alloy elements molybdenum, chromium and vanadium forms hard and stable carbides with the excess carbon resulting from the transformed martensitic phase. The hard carbides precipitate in the remaining martensitic phase of the connectors and compensate thereby for the hardness, that is lost by transformation of martensite into cementite.

[0016] The core of the connectors consists of martensite and bainite. Bainite is a fine mixture of the phases cementite and ferrite. Bainite is stable at high temperatures and remains therefore sufficiently strong to support the hardened surface zone of the connectors at high working temperatures.

[0017] According to an alternative, the amount of Si is 0.85 - 0.95 wt% in the inventive steel.

[0018] According to an alternative, the amount of Mo is 0.70 - 0.80 wt% in the inventive steel.

[0019] According to an alternative, the amount of Cr is 1.20 - 1.25 wt% in the inventive steel.

[0020] According to an alternative, the amount of V is 0.20 - 0.30 wt%, preferably 0.2 - 0.25 wt% in the inventive steel.

[0021] According to an alternative, the amount of N is 0.005 - 0.008 wt% more preferred 0.008 - 0.012 wt%, in the inventive steel.

[0022] The invention also relates to component for rock drilling comprising the inventive steel.

[0023] The component may be a threaded male or female connector for a drill rod.

[0024] For example, the component is a drill rod comprising a threaded male and a threaded female connector.

[0025] The invention also relates to a method for manufacturing a component for rock drilling comprising the steps of:

- a. forming a component for rock drilling as described above from the inventive steel.
- b. heating said component to austenitizing temperature;
- c. holding said component at austenitizing temperature in a carbon containing atmosphere for a predetermined time;
- d. cooling said component.

[0026] Preferably, said component is heated to a temperature of 900 - 1000°C.

[0027] Preferably, said component is heated in an atmosphere of CO and H₂.

[0028] Preferably, the component is heated for 3-6 hours.

[0029] Preferably, the component is cooled in air.

[0030] The invention also relates to the use of the inventive bainitic steel in case hardened connectors for drill rods during air cold top hammer drilling above ground.

DETAILED DESCRIPTION OF THE INVENTION

[0031] The inventive steel comprises the following elements in weight% (wt%):

[0032] Carbon (C). Carbon is included in the inventive steel for strength and to govern the final structure of the steel, which should be bainitic. Carbon is also added to the inventive steel for ensuring the formation of carbides. The carbides provide a precipitation hardening effect in the bainitic structure of the steel. The carbides further prevent the grains in the steel from growing by coalescence, and thereby ensures fine grains in the steel and consequently high strength. The carbon content should therefore be at least 0.16 wt% in the steel. Too high carbon content reduces the impact strength of the steel. Carbon should therefore be limited to 0.23 wt%. Preferably, carbon is 0.18 - 0.20 wt%.

[0033] Silicon (Si) is used as deoxidizer in the manufacturing of the steel and some amounts of silicon is therefore always present in the steel. Silicon has a positive effect on the inventive steel since it increases the hardenability, i.e. the rate by which the austenitic phase is transformed into martensite during quenching. In the inventive steel, silicon is an important alloy element since it retards the transformation of martensite into cementite and ferrite.

[0034] Martensite is an unstable phase and when heated it transforms, via various carbides, into cementite and ferrite which leads to decreased hardness of the steel. Silicon stabilizes epsilon carbide, which is one of the carbides that precedes the cementite phase during the transformation of martensite and thereby retards the transformation of martensite. Furthermore, during the dissolving of the martensitic phase, carbon must diffuse through the steel to the carbides in order for the carbides to grow. The presence of silicon in the steel increases the carbon activity in the steel which in turn retards the growth of the already formed carbides and also the nucleation of new carbides. Also this mechanism substantially retards the transformation of the martensite. Silicon has therefore a positive effect on retaining the strength of the surface zone in case hardened components of the inventive steel at high temperatures.

[0035] However, silicon stabilizes ferrite and therefore will too high amounts of silicon lead to an increase of the A1-temperature. This has a negative effect as the steel during hardening must be heated to higher temperature which causes grain growth in the austenitic phase and thereby reduces the strength. Consequently, the amount of silicon is limited to 0.80 - 1.0 wt% in the inventive steel. Preferably, the amount of silicon is 0.85 - 0.95 wt%.

[0036] Molybdenum, chromium and vanadium are key elements in the inventive steel since they form hard carbides which compensate for the hardness drop when the martensitic phase transforms into cementite and ferrite. The different carbide formers molybdenum, chromium and vanadium form stable carbides at various temperatures. Hence, at low temperatures and therefore moderate transformation of the martensite, mainly molybdenum rich carbides are precipitated. With increasing temperatures the transformation of martensite increases. However at higher temperatures, chromium rich carbides are first precipitated and subsequently, at even higher temperatures, also vanadium rich carbides. This provides the effect that the hardness of the martensite in the surface of the connector is kept substantially constant over a wide range of working temperatures.

[0037] Molybdenum (Mo), forms stable molybdenum rich carbides at a temperature from 300°C up to approximately 500°C and compensates for the hardness drop when the martensitic phase is transformed into cementite and ferrite. To ensure that a sufficient amount of carbides is precipitated, the amount of molybdenum shall be at least 0.67 wt%. However, molybdenum stabilizes austenite and has therefore a very strong influence on hardenability. Too high amounts of molybdenum could therefore lead to the formation of martensite in the core of the connector, which make the connector brittle. High amounts of molybdenum could also cause the formation of secondary hardness maximum. The upper limit for molybdenum is therefore 0.9 wt% in the inventive steel. Preferably, molybdenum is 0.67 to 0.83 wt% in the steel.

[0038] Chromium (Cr) forms stable chromium rich carbides with carbon. Some chromium rich carbides are precipitated even at low temperatures, i.e. 300°C. However, the majority of the chromium rich carbides are precipitated at temperature between 400 - 500°C. To ensure that a sufficient amount of chromium rich carbides are formed, the inventive steel should contain at least 1.10 wt% chromium. Very high amounts of chromium could lead to the formation of a so called secondary hardness maximum in the steel at high temperatures, typically above 600°C. This phenomenon is generally caused by the formation of a large amount of chromium carbides, and also of vanadium- and molybdenum carbides. However, if the temperature of the steel is increased further, the hardness rapidly drops due to growth of the precipitated carbides which in turn steal carbon from other precipitations in the steel. Chromium should therefore be limited to 1.30 wt%. Preferably, the content of chromium is 1.20 - 1.25 in the inventive steel to ensure that sufficient amount of carbides are formed and that the formation of a secondary hardness maximum is avoided.

[0039] Vanadium (V) form very small vanadium rich carbides at temperatures of 550 - 600°C and compensate therefore for the hardness drop when the martensitic phase transforms into cementite and ferrite at high temperatures. The

inventive steel should contain at least 0.18 wt% vanadium to ensure that a sufficient amount of vanadium carbides is precipitated in the steel at high working temperatures.

[0040] Vanadium also forms vanadium carbonitrides at high temperatures, i.e. 900°C and above. The vanadium carbonitrides are important since they prevent grain growth of the austenitic phase during carburization of the steel. Too high amounts of vanadium could lead to problems during hot working of the steel since the carbonitrides becomes so stable that they do not dissolve in the annealing step that precedes hot working. Therefore vanadium must be limited to 0.40 wt% in the inventive steel. Preferably, vanadium is 0.18 - 0.30 wt%, more preferred 0.20 - 0.30 wt%, even more preferred 0.20 - 0.25 wt%.

[0041] Manganese (Mn) is included in the inventive steel for forming MnS with sulphur, which may be present as an impurity in the steel. Manganese has a positive effect on hardenability of the steel, since it lowers the Ms-temperature, i.e. the temperature at which martensite start to form after austenitizing. The low Ms-temperature also causes a fine bainitic structure in the core of a connector manufactured from the inventive steel. This is positive for ensuring a high strength in the core of the connector. Manganese should be included in an amount of at least 0.65 wt% in order to ensure MnS-types of sulfides. High amounts of manganese could result in the formation of retained austenite in the steel, due to that manganese lowers the Ms-temperature. Manganese should therefore be limited to 0.85 wt%. Preferably the amount of manganese is 0.70 - 0.80 wt% in the steel since this amount of manganese also ensures a fine bainitic structure in the inventive steel.

[0042] Phosphorus (P) is present as an impurity in the raw material for the inventive steel. Phosphorous segregate to the liquid phase during solidification of the steel and causes phosphorous rich streaks in the solidified steel. A high phosphorous content therefore has a negative impact on the ductility and impact toughness of the steel. Therefore, phosphor should be limited to a maximum of 0.020 wt%, i.e. 0 - 0.020 wt%, in the inventive steel.

[0043] Sulphur (S) is also present as an impurity in the raw material for the inventive steel. Sulphur forms sulphide inclusions in the steel which has a negative impact on the ductility and impact strength of the steel. Sulphur should therefore be limited to 0.02wt%, i.e. 0 - 0.020 wt%, in the inventive steel, more preferred to max 0.015 wt%.

[0044] Nickel (Ni) increases the impact strength of the steel and is consequently an important element in the inventive steel which is intended for drilling rods. Nickel further reduces the Ms-temperature of the steel and increases thereby the hardenability. In order to ensure sufficient impact strength in the steel, the nickel content should be at least 1.60 wt%. Too high content of nickel could reduce the Ms-temperature too much and lead to the formation of retained austenite in the steel. Retained austenite could cause tensile stress in the martensitic phase, and thereby reduce the strength of the steel. The nickel content should therefore be limited to 2.0 wt% in the inventive steel. Nickel is further an expensive alloying element and should for that reason be present in as low amounts as possible. Preferably, the content of nickel is 1.70 - 1.90 wt% in the inventive steel since this amount of nickel yields a cost effective steel with sufficient impact strength.

[0045] Copper (Cu) is typically included in the scrap metal that is used as raw material. Copper may be allowed in amounts up to 0.20 wt%, i.e. 0-0.20 wt%.

[0046] Nitrogen (N). The inventive steel preferably contains nitrogen to ensure that the stable vanadium carbonitrides are formed during carburization. Preferably, the amount of nitrogen is 0.005 wt%, more preferred 0.008 wt%. If the steel contains too much nitrogen, the vanadium carbonitrides will become too stable and may not dissolve during heating to the hot working temperature of the steel. Therefore the maximum amount of nitrogen is 0.012 wt%.

[0047] In hot rolled condition, the inventive steel has a throughout bainitic structure, i.e. a structure of cementite (Fe_3C) and ferrite (α -iron). By "hot rolled" is meant that the inventive steel has been produced by casting, thereafter been heated to a temperature of approximately 1200°C and subjected to hot rolling followed by cooling in air.

[0048] In case hardened condition, the inventive steel has a martensitic surface zone and a bainitic/martensitic core.

BRIEF DESCRIPTION OF THE DRAWINGS

[0049]

Figure 1: A schematic drawing of a rock drilling component manufactured comprising the inventive steel.

Figure 2: A graph showing the results from experiments performed on the inventive steel.

Figure 3: A table showing the results from tests performed on the inventive steel.

Figure 4 and 5: Surface and core hardness of samples in a test performed on an inventive steel and a comparative steel.

Figure 6 to 10: Diagrams produced in ThermoCalc™ simulations performed on an inventive and a comparative steel.

DESCRIPTION OF EMBODIMENTS

[0050] Figure 1 shows schematically a longitudinal cross-section of a drilling component according to a first embodiment of the present invention. The drilling component shown in figure 1 is a MF-drilling rod 1, which comprises a central rod portion 10. The first end of the central rod 10 comprises a male connector 20 and the second end of the central rod comprises a female connector 30. The male connector 20 is provided with an external thread 21 and the female connector is provided with an internal thread 31. The dimensions of the male and the female connectors and the threads 21, 31 are dimensioned such that the male connector 20 of a first MF rod can be received in the female connector 30 of a second MF-rod. The MF-rod further comprises a central channel 60, i.e. a bore that extends through the entire MF-rod. The channel has one opening 61 in the center of the male connector and one opening 61 in the centre of the female connector. In operation, cooling fluid, such as air is lead through the channel 60.

[0051] In figure 1, the male and the female connectors 20, 30 are attached to the central rod portion 10 by friction welding which is indicated by the dashed lines 11. However, the MF-rod in figure 1 could also be manufactured in one piece, i.e the male and the female connectors 20 and 30 could be formed by forging and threading the ends of the rod.

[0052] The connectors 20 and 30 are manufactured from the bainitic steel according to the invention. The central rod 10 may be manufactured from another type of steel, for example a conventional low-alloyed carbon steel. However, the central rod could also be manufactures from the bainitic steel according to the invention.

[0053] The connectors 20 and 30 are case hardened and have a bainitic core 40 and a martensitic surface zone 50. The martensitic surface zone is 1 - 3 mm thick and extends from the surface of the connector towards its centre.

[0054] Although the inventive drilling component has been described with regards to a MF-rod it is obvious that it also could be any other type of component that is subjected to repeated wear under high working temperatures, for example a drifter rod.

[0055] Preferably, the inventive drilling component is manufactured by a method which comprises the following steps.

[0056] In a first step, a drilling component is formed in a bainitic steel according to the invention. This is typically achieved by forging and threading a precursor of the inventive steel into male and female connectors 20, 30. The precursor is typically a portion of a solid rod that has been manufactured from the inventive steel.

[0057] In a second step, the connectors are subjected to case hardening. This is achieved in that the connectors are heated in a furnace to austenitizing temperature, which for the inventive steel is above 900°C. The furnace could be of any type, e.g a pit furnace. In order to ensure complete austenitizing of the connectors and to avoid negative effects, such as grain enlargement, the connectors should be heated to temperature between 900°C and 950°C, preferably 925°C.

[0058] The step of austenitizing of the connectors is performed in a carbon rich atmosphere to ensure that the content of carbon is increase in the surface zone of the connectors, so called carburization. Typically the atmosphere in the furnace is a mixture of the gases H₂ and CO, for example cracked methane.

[0059] The connectors are kept in the furnace for a time period of 3 - 6 hours. The time governs the case depth, i.e. the thickness of the martensitic surface zone. Preferably the time period is 5 hours to ensure a sufficient case depth.

[0060] When the heating time has expired, the connectors, which now are austenitized, are taken out of the furnace and are cooled in the ambient air. Forced air cooling may be employed by blowing air onto the connectors.

[0061] During cooling the carburized surface of the austenitized connectors transforms into martensite and the core of the connectors into a mixture of bainite and martensite.

[0062] The connectors may thereafter be subjected to a tempering step to optimized the hardness of the martenistic surface. Tempering is thereby performed at 200 - 300 °C for 1 hour.

[0063] Finally, the connectors are attached to a central rod portion by friction welding.

EXAMPLES

[0064] The inventive steel material is following described by four non-limitating examples.

Example 1

[0065] Example 1 describes the results from field tests performed with case hardened drill rods manufactured from the inventive bainitic steel.

[0066] In a first step a heat of the inventive steel was produced. The heat was produced by melting scrap metal in an electric arc furnace, refining of the molten steel in a CLU converter and subsequently cast in 24" moulds to ingots.

[0067] The obtained inventive steel had the following composition:

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Table 1: Chemical composition of inventive steel

C	Si	Mn	P	S	Cr	Ni	Mo	V	Cu	N
0.19	0.87	0.72	0.004	0.009	1.15	1.66	0.70	0.20	0.13	0.009

[0068] From the inventive steel rods were produced. Some of the rods were forged into threaded female type connectors and some into threaded male type connectors.

[0069] The male and female type connectors were subjected to case hardening. In a first step the connectors were carburized in a pit furnace at a temperature of 925 °C for a time period of 5 hours, the furnace contained an atmosphere of CO and H₂.

[0070] After five hours the connectors were removed from the furnace and allowed to cool in air. The case hardening resulted in a martensitic layer which extended from the surface of the connector towards the core which had bainitic/martensitic structure.

[0071] The connectors were thereafter attached to the end of a steel rod which also was manufactured from the inventive steel material. A male connector was attached to one end of the rod and a female connector to the other end. The connectors were attached by friction welding.

[0072] Field testing was thereafter performed with the drilling rods from the inventive steel at two different locations, Site A and Site B. Drilling was performed with a drill bit having a diameter of 115 mm and a drilling rig of the type Sandvik DP1500 was used. The drilling speed was approximately 1 meter/minute.

[0073] As comparison were also conventional drill rods used. These rods were made of the steel grade Sanbar 64.

[0074] Nine rods of each type (inventive and conventional) were used at Site A and 4 rods of each type at site B. The drill rods were used until failure and the total number of meters drilled with each rod was recorded as "drilling meter (dm)". Table 2 shows the result of the testing as the average number of drilling meters drilled per rod at site A and at site B.

Table 2: Results from drilling

Site	Conventional rod	Inventive rod
Site A	2400 dm (average)	3200 dm (average)
Site B	2100 dm (average)	3100 dm (average)

[0075] As can be seen in table 1, the drilling rods of the inventive steel had a considerable longer operational life length than the rods of the conventional material.

Example 2

[0076] In a second example, the hardness reduction of test samples from an inventive steel was determined under laboratory conditions at various reheating temperatures.

[0077] In a first step, a heat of the inventive steel was produced. The heat was produced by melting scrap metal in an electric arc furnace, refining of the molten steel in a CLU converter and subsequently casting in 24" moulds to ingots.

[0078] The obtained inventive steel had the following composition:

Table 3: Chemical composition of inventive steel

C	Si	Mn	P	S	Cr	Ni	Mo	V	Cu	N
0.20	0.89	0.79	0.011	0.013	1.27	1.75	0.77	0.21	<0.01	0.008

[0079] The ingots were rolled into bars and the bars were cut into 5 cm long cylinders, which were used as samples.

[0080] The samples were thereafter subjected to a simulated hardening treatment. This treatment included heating to austenitizing temperature, holding at austenitizing temperature for a pre-determined temperature and subsequently cooling in oil which was heated to room temperature. Thereafter the hardened samples were subjected to reheating in order to simulate heating during drilling operation. After reheating, the samples were cooled in air. After cooling of the reheated samples, the hardness was measured in the surface, on the middle of the radius and in the center of each sample. The hardness was measured in Vickers (HV1)

[0081] As reference, one sample of each series was left as hardened but in non reheated condition.

[0082] Twelve samples were used for each austenitizing temperature. The austenitizing temperatures was :860°C, 1 h holding time; 880°C, 1 h holding time; 925°C, 20 min holding time. After quenching in oil, the samples were reheated

at the following temperatures: Non Reheated, 200°C, 300°C, 400°C, 500°C, 550°C, 580°C, 600°C, 650°C, 675°C and 700°C.

[0083] The result of the measurement graphically demonstrated in figure 2. Figure 2 shows a graph in which the result for each austenitizing temperature is shown as a mean value for the measured hardness at each reheating temperature. The specific measurement values are shown in table 4, see figure 3.

[0084] It should be noted that the experiment is performed on non-carburized samples. However, from graph in figure 2, it is clear that the hardness of the three different samples series is almost constant from the non-reheated samples up to 650°C. It is believed that the constant hardness is due to the stabilizing effect of silicon on the martensitic phase at low temperatures and by the precipitation of hard and stable carbides of chromium, molybdenum and vanadium at higher temperatures which compensates for the transformation of martensite into cementite and ferrite. At 700°C, a secondary hardness maximum is formed and thereafter the hardness sharply drops due to that the Cr-, Mo- and V-carbides coalesce into fewer and coarser precipitations. The growth of the Cr-, Mo- and V-carbides further causes the remaining martensite to dissolve into cementite and ferrite and thereby the hardness decreases even further.

[0085] It is evident that a carburized sample of the inventive steel material, at all reheating temperatures, would be harder than the non-carburized samples. However, it is believed that the hardness of a carburized sample would also exhibit an essentially constant hardness up to approximately 650°C.

Example 3

[0086] In a third example a comparison was made on the surface- and core hardness of hardened and tempered samples of an alloy according to the invention and a comparative alloy. The test simulates the tempering effect that occurs in case hardened drill rods due to the heat that evolves in the couplings during drilling. For comparison, an alloy similar to the alloy disclosed in document WO97/27022 was selected. WO97/27022, discloses an alloy which is optimized for friction welding and is briefly discussed under the section "Background of the invention" of the present application.

[0087] The chemical composition of the inventive and comparative alloys are shown in table 5 below. Comp 0.09 denominates the comparative alloy and Inv 0.22 denominates the inventive alloy.

Table 5: Chemical composition of test alloys

	%C	%Si	%Mn	%P	%S	%Cr	%Ni	%Mo	%V	%Cu	%N
Comp 0.09V	0.19	0.89	0.30	0.005	0.002	1.25	1.79	0.75	0.09	0.020	0.002
Inv 0.22V	0.20	0.89	0.70	0.060	0.027	1.20	1.84	0.70	0.22	0.13	0.009

[0088] A 1 kg heat of the comparative alloy was produced by conventional methods including: melting of scrap metal in a induction furnace, refining and casting. The casting was preheated in a furnace in 700°C for approx. 30 minutes and then hot rolled at 1200°C into a square bar having the dimensions 13 mm. The bar was then slowly cooled in air and cut into 13 x 13 mm samples.

[0089] A 75 ton heat of the inventive alloy was produced by conventional methods used in production, including: melting in an EA-furnace, AoD treatment, ladle refining, continuous casting and hot rolling. The obtained casting of the inventive material was hot rolled to a bar having a diameter of 40 mm.

[0090] The bars of the inventive material were cut into samples in dimensions 40 x 130 mm.

[0091] The samples were subsequently carburized and hardened by forced air cooling. Carburizing of the samples was performed according to the following program in an atmosphere of Propane/Nitrogen/Methanol. In Step 1 the samples were first heated for a period of 150 minutes to the process temperature of 925°C and then held at that temperature for 435 min:

Table 6: Carburizing program

	Step 1	Step 2	Step 3
Temperature, °C	925	925	925
Carbon potential (Cp)	0.80	0.60	0.40
Time, min	150	0	0
Hold time, min	435	100	180

[0092] Thereafter, the hardened samples were subjected to tempering at different temperatures. Prior to tempering,

the samples were painted with No-Carb™ in order to prevent decarburization. Table 7 below shows the tempering temperature for each sample. one sample of each alloy was left untempered. Each of the remaining samples was tempered for 30 minutes.

Table 7: Tempering temperatures

Sample	1	2	3	4	5	6	7	8	9	10
Temperature, °C	Untempered	150	180	200	250	300	400	500	600	700

[0093] After tempering, the core and surface hardness of each sample were measured. The surface hardness was measured in HRC and the core hardness by Vickers measurement (HV30). The surface hardness of the various samples is shown in figure 4. The core hardness of the samples is shown in figure 5.

[0094] From figure 4 it can be concluded that the untempered samples of the inventive and the comparative alloy have similar surface hardness. This is due to that the structure in the surface of the respective untempered samples essentially consists of martensite. The hardness of the tempered samples decreases with increasing tempering temperature. However, from the graphs in figure 4 it is clearly visible that the surface hardness of the inventive alloy is higher than the surface hardness of the comparative alloy for all tempering temperatures up to 600°C. That is, the inventive alloy has a higher tempering resistance than the comparative alloy..

[0095] Surprisingly, the surface hardness of the inventive alloy remains much more stable with increasing tempering temperature than the surface hardness of the comparative alloy. As can be seen in figure 4, the surface hardness of the inventive alloy is essentially constant at 57 HRC up to 200°C where it drops to 55 HRC and then proceeds essentially constant up to 300°C. The surface hardness of the comparative alloy on the other hand drops continuously over the whole temperature interval.

[0096] At higher temperatures the dissolving rate of the martensite increases and the vanadium carbides coalesce to coarser particles which results in decreasing surface hardness. At 700°C the vanadium carbides become unstable and the surface hardness of both the inventive and the comparative samples drops rapidly.

[0097] From figure 5 it can be concluded that the core hardness in the inventive samples is slightly lower than in the comparative samples. The main reason for the relative low core hardness of the inventive alloy is that the high amount of vanadium in combination with the selected nitrogen content produces stable vanadium carbonitrides during the carburizing step of the samples. The small vanadium carbonitrides prevents grain growth during the carburizing step and increases the impact toughness of the core. The small grains also lowers the hardenability of the alloy and ensures thereby that the core, after hardening, substantially consists of bainite which is less hard but more tough than martensite.

Conclusion

[0098] The results from the third example show a better tempering resistance in the inventive alloy than in the comparative alloy. The surface hardness of the inventive alloy is more stable compared with the comparative material.

[0099] In rock drilling, the ability to have a stable surface hardness is crucial for the wear resistance. A material that will keep the surface hardness even though the temperature increases during drilling will withstand wear better, as adhesive wear resistance is in direct relation with the hardness. The relation between surface hardness and core hardness is also an important factor for threads used in drilling rods. The desired relation is a hard surface for better wear resistance together with a tough core for better impact resistance. Also a greater difference between hardness of the surface and the core results in more residual compressive stresses, which increases fatigue life. With this in mind the inventive alloy with high vanadium content is advantageous compared with the comparative material having a low vanadium content, it provides a higher surface hardness together with a tougher core, while it is the opposite for the comparative material.

Example 4

[0100] In a fourth example, simulations were performed in the program ThermoCalc™ 3.0 and database TCFE7. The purpose of the simulations was to confirm the results from the measurements of the core hardness on the inventive and the comparative samples in the third example. A further purpose was to confirm that the good result of core hardness of the inventive sample exist over a preferred range of nitrogen and vanadium of the inventive alloy.

[0101] The simulations shows the stability of vanadium carbonitrides at various temperatures in inventive and comparative alloys. As will be described further below, the presence of vanadium carbonitrides at the carburizing temperature or the hotworking temperature will have a significant effect on the metallographic structure in the core a final component.

[0102] Figure 6 shows a diagram produced in a first ThermoCalc™ simulation of the stability of vanadium carbonitrides that are formed in an inventive alloy having a vanadium content of 0.2 wt% and a nitrogen content of 0.005 wt%. The

overall composition of the alloy in the simulation is:

0.019 C; 0.9 Si; 0.75 Mo; 1.2 Cr; 0.20 V; 1.8 Ni; 0.78 Mn; 0.005 N

[0103] Figure 6 shows the amount of various precipitated phases in moles that exist in the alloy system at different temperatures. The y-axis shows the amount of precipitated phases and the x-axis shows the temperature. Line 1 shows the amount (in moles) of vanadium carbonitrides that exists in the alloy system at various temperatures. The other lines shows in the diagram shows other phases that are present in the inventive alloy system. These phases will not be discussed further.

[0104] When line 1 is followed in figure 6, it can be seen that the precipitation of vanadium carbonitrides increases with increasing temperature in the temperature range of 700 - 800°C. Above 800°C the precipitation of vanadium carbonitrides ceases and the precipitated vanadium carbonitrides start to dissolve due to equilibria in the alloy system. Consequently, less vanadium carbonitrides may exist in the alloy system at high temperatures. The amount of carbonitrides in the alloy system therefore decreases with increasing temperature. In the alloy system of figure 6 it can be seen that a relatively high amount of vanadium carbonitrides exists in the alloy system in the temperature interval of 900 - 1000°C. The diagram further shows that the vanadium carbonitrides are entirely dissolved at approx. 1100°C.

[0105] The above distribution of vanadium carbonitrides would ensure good core properties in a component manufactured from the inventive alloy for the following reasons:

[0106] Firstly, in production of components for rock drilling, the components are carburized and hardened at 930°C. At this temperature the crystal grains in the steel strive to coalesce into few and large grains.

[0107] Generally, the grain size of a steel influences the hardenability of the steel in the sense that the hardenability of the steel increases with increasing grain size. After hardening, a steel with a small grain size will therefore, have a predominant bainitic structure whereas a steel with large grains will have a martensitic structure.

[0108] The presence of the relatively large amount of vanadium carbonitrides at 930°C in figure 6 would effectively prevent grain growth in the inventive steel by blocking the crystal grains of the alloy from coalescing. This would in turn result in small grains in the inventive alloy and a predominately bainitic structure in the core of a hardened component manufactured thereof. This is important for the strength and impact toughness of the core as well as its structural stability at high temperature.

[0109] Secondly, from figure 6 it may be concluded that all vanadium carbonitrides are dissolved at approx. 1100°C. This is of course important for the hotworkability of the steel. However, more important is the absence of the negative effect that vanadium carbonitrides remaining after hotworking would have on the grain size during hardening of the alloy. In the hardening step remaining vanadium carbonitrides would coalesce into few and very large particles. These particles would have little effect on preventing grain growth during carburization/hardening and the result would be a component with a core of mainly martensitic structure having low toughness and therefore poor impact strength.

[0110] Figure 7 shows a diagram produced in a second ThermoCalc™ simulation of the stability of vanadium carbonitrides that are formed in an inventive alloy with a vanadium content of 0.2 and a nitrogen content of 0.012. This simulation confirms the conclusions of the first simulation. Hence, also this simulation shows that a sufficient amount of vanadium carbonitrides exist in the alloy in the temperature interval of 900 - 1000°C to ensure a bainitic structure in the core of the alloy after hardening. It may further be concluded from the diagram that the vanadium carbonitrides are completely dissolved at approx. 1130°C.

[0111] It can be noted that the higher nitrogen content in the alloy of the second simulation results in the precipitation of more vanadium carbonitrides at 930°C in comparison with the first simulation. This is of course positive for ensuring the bainitic structure of the core.

[0112] Figure 8 shows a diagram produced in a third ThermoCalc™ simulation of the stability of vanadium carbonitrides that are formed in an inventive alloy with a vanadium content of 0.3 wt% and a nitrogen content of 0.005 wt%. The simulated alloy had the following composition:

0.019 C; 0.9 Si; 0.75 Mo; 1.2 Cr; 0.1 V; 1.8 Ni; 0.78 Mn; 0.005 N

[0113] Also this simulation shows that a sufficient amount of vanadium carbonitrides are precipitated at 900-1000°C and that all vanadium carbonitrides have dissolved at a temperature of 1120°C.

[0114] In comparison to the first and second simulations more vanadium carbonitrides are precipitated in the third simulation. The reason for this is the higher vanadium content in this alloy.

[0115] Figure 9 shows a diagram produced in a fourth ThermoCalc™ simulation of the stability of vanadium carbonitrides that are formed in an inventive alloy with a vanadium content of 0.3 wt% and a nitrogen content of 0.012 wt%. The simulated alloy had the following composition:

0.019 C; 0.9 Si; 0.75 Mo; 1.2 Cr; 0.1 V; 1.8 Ni; 0.78 Mn; 0.005 N

[0116] Also this simulation shows that a sufficient amount of vanadium carbonitrides exists at the temperature range of 900-1000°C and that the vanadium carbonitrides have dissolved at a temperature below 1200°C.

[0117] Figure 10 shows a diagram produced in a fifth ThermoCalc™ simulation of the stability of vanadium carbonitrides that are formed in a comparative alloy with low vanadium content (0.1 wt%) and a nitrogen content of 0.005 wt%. The simulated alloy is similar to the alloy used in Example 3 and has the following composition:

0.019 C; 0.9 Si; 0.75 Mo; 1.2 Cr; 0.1 V; 1.8 Ni; 0.78 Mn; 0.005 N

[0118] From line 1 in figure 10 it can be concluded that a very small amount of vanadium carbonitrides exist in this alloy at the temperature interval of 900 - 1000°C. In this alloy the amount of vanadium carbonitrides is too small to prevent grain growth during carburization which in turn would result in increased hardenability and martensite formation in the core of a hardened component manufactured this alloy. The simulation therefore confirms the measurements that was made on the core hardness of the comparative alloy of Example 3.

[0119] To summarize, from the five ThermoCalc™ simulations and the results from the physical experiment 3 it may be concluded that an optimal balance of surface hardness and core hardness is achieved in the inventive alloy. The optimal balance of surface- and core hardness makes the inventive alloy very suitable for use in rockdrilling components.

Claims

1. Bainitic steel comprising, in weight% (wt%):

C:	0.16 - 0.23
Si:	0.8 - 1.0
Mo:	0.67 - 0.9
Cr:	1.10 - 1.30
V:	0.18 - 0.4
Ni:	1.60 - 2.0
Mn:	0.65 - 0.9
P:	≤ 0.020
S:	≤ 0.02
Cu:	< 0.20
N:	0.005 - 0.012 wt%

balance Fe and unavoidable impurities.

2. The bainitic steel according to claim 1, wherein the amount of Si is 0.85 - 0.95 wt%.

3. The bainitic steel according to claim 1 or 2, wherein the amount of Mo is 0.70 - 0.80 wt%.

4. The bainitic steel according to any of claims 1 - 3, wherein the amount of Cr is 1.20 - 1.25 wt%.

5. The bainitic steel according to any of claims 1 - 4, wherein the amount of V is 0.20 - 0.30 wt%, preferably 0.2 - 0.25 wt%.

6. The bainitic steel according to any of claims 1 - 5, wherein the amount of N is 0.008 - 0.012 wt%.

7. A component for rock drilling (10, 20, 30) comprising a steel according to any of claims 1 - 6.

8. The component according to claim 7, wherein the component is a threaded male or female connector (20, 30) for a drill rod (10).

9. The component according to any of claims 7 or 8, wherein the component is a drill rod (10) comprising a threaded male and a threaded female connector (20, 30).

10. Method for manufacturing a component for rock drilling (10, 20, 30) comprising the steps of:

- a. forming a component (10, 20, 30) for rock drilling according to any of claims 7 - 9 in a steel according to any of claims 1 - 6;
- b. heating said component (10, 20, 30) to austenitizing temperature;
- c. holding said component (10, 20, 30) at austenitizing temperature in a carbon containing atmosphere for a predetermined time;
- d. cooling said component.

11. The method according to claim 10, wherein the component is heated to a temperature of 900 - 1000°C.

12. The method according to claim 10 or 11, wherein the component is heated in an atmosphere of CO and H₂.

13. The method according to any of claims 10 - 12 wherein the component is heated for 3-6 hours.

14. The method according to any of claims 10 - 13, wherein the component is cooled in air.

15. Use of a bainitic steel according to any of claims 1 - 6 in case hardened connectors for drill rods during air-cold top hammer drilling above ground.

Patentansprüche

1. Bainitstahl, der in Gewichts.-% (Gew.-%):

C:	0.16-0.23
Si:	0.8 - 1.0
Mo:	0.67-0.9
Cr:	1.10 - 1.30
V:	0.18-0.4
Ni:	1.60-2.0
Mn:	0.65-0.9
P:	≤0.020
S:	≤0.02
Cu:	< 0.20
N:	0.005 - 0.012 Gew.-%,

und im Übrigen Eisen sowie unvermeidbare Verunreinigungen enthält.

2. Bainitstahl nach Anspruch 1, wobei der Anteil an Si 0.85 - 0.95 Gew.-% beträgt.

3. Bainitstahl nach Anspruch 1 oder 2, wobei der Anteil an Mo 0.70 - 0.80 Gew.-% beträgt.

4. Bainitstahl nach einem der Ansprüche 1-3, wobei der Anteil an Cr 1.20 - 1.25 Gew.-% beträgt.

5. Bainitstahl nach einem der Ansprüche 1-4, wobei der Anteil an V 0.20 - 0.30 Gew.-%, vorzugsweise 0.2 - 0.25 Gew.-%, beträgt.

6. Bainitstahl nach einem der Ansprüche 1-5, wobei der Anteil an N 0.008 - 0.012 Gew.-% beträgt.

7. Bauteil für das Gesteinsbohren (10,20,30), das einen Stahl nach einem der Ansprüche 1-6 enthält.

8. Bauteil nach Anspruch 7, wobei das Bauteil ein Außengewindeanschluss oder Innengewindeanschluss (20, 30) für eine Bohrstange (10) ist.

9. Bauteil nach einem der Ansprüche 7 oder 8, wobei das Bauteil eine Bohrstange (10) ist, die einen Außengewindeanschluss und einen Innengewindeanschluss (20, 30) aufweist

10. Verfahren zur Herstellung eines Bauteils für das Gesteinsbohren (10, 20, 30), das folgende Schritte aufweist:

- a) Herstellen eines Bauteils (10, 20, 30) für das Gesteinsbohren nach einem der Ansprüche 7-9 aus einem Stahl nach einem der Ansprüche 1-6;
- b) Aufheizen des Bauteils (10, 20, 30) bis zur Austenitisierungstemperatur;
- c) Halten des Bauteils (10, 20, 30) für eine vorbestimmte Zeit bei der Austenitisierungstemperatur in einer kohlenstoffhaltigen Atmosphäre
- d) Abkühlen des Bauteils

11. Verfahren nach Anspruch 10, wobei das Bauteil auf eine Temperatur von 900 - 1000°C erhitzt wird.

12. Verfahren nach Anspruch 10 oder 11, wobei das Bauteil in einer Atmosphäre aus CO und H₂ erhitzt wird.

13. Verfahren nach einem der Ansprüche 10-12, wobei das Bauteil für 3-6 Stunden erhitzt wird.

14. Verfahren nach einem der Ansprüche 10-13, wobei das Bauteil an Luft gekühlt wird.

15. Verwendung eines Bainitstahl nach einem der Ansprüche 1-6 für gehärtete Gewinde von Bohrstangen beim luftgekühlten Tophammerbohren über Tage.

Revendications

1. Acier bainitique comprenant, en % en poids :

C :	de 0,16 à 0,23
Si :	de 0,8 à 1,0
Mo:	de 0,67 à 0,9
Cr:	de 1,10 à 1,30
V:	de 0,18 à 0,4
Ni:	de 1,60 à 2,0
Mn:	de 0,65 à 0,9
P:	≤ 0,020
S:	≤ 0,02
Cu:	< 0,20
N :	de 0,005 à 0,012 % en poids

le reste étant du Fe et des impuretés inévitables.

2. Acier bainitique selon la revendication 1, dans lequel la proportion de Si est comprise entre 0,85 et 0,95 % en poids.

3. Acier bainitique selon la revendication 1 ou 2, dans lequel la proportion de Mo est comprise entre 0,70 et 0,80 % en poids.

4. Acier bainitique selon l'une quelconque des revendications 1 à 3, dans lequel la proportion de Cr est comprise entre 1,20 et 1,25 % en poids.

5. Acier bainitique selon l'une quelconque des revendications 1 à 4, dans lequel la proportion de V est comprise entre 0,20 et 0,30 % en poids, de préférence entre 0,2 et 0,25 % en poids.

6. Acier bainitique selon l'une quelconque des revendications 1 à 5, dans lequel la proportion de N est comprise entre 0,008 et 0,012 % en poids.

7. Composant pour le forage de roches (10, 20, 30) comprenant un acier selon l'une quelconque des revendications 1 à 6.

8. Composant selon la revendication 7, où le composant est un raccord mâle ou femelle fileté (20, 30) destiné à une tige de forage (10).

5 9. Composant selon l'une quelconque des revendications 7 ou 8, où le composant est une tige de forage (10) comprenant un raccord mâle fileté et un raccord femelle fileté (20, 30).

10. Procédé de fabrication d'un composant pour le forage de roches (10, 20, 30) comprenant les étapes de :

- 10 a. formation d'un composant (10, 20, 30) pour le forage de roches selon l'une quelconque des revendications 7 à 9 dans un acier selon l'une quelconque des revendications 1 à 6 ;
b. chauffage dudit composant (10, 20, 30) à une température d'austénitisation ;
c. maintien dudit composant (10, 20, 30) à la température d'austénitisation dans une atmosphère contenant du carbone pendant une période prédéterminée ;
15 d. refroidissement dudit composant.

11. Procédé selon la revendication 10, dans lequel le composant est chauffé à une température comprise entre 900 et 1 000 °C.

20 12. Procédé selon la revendication 10 ou 11, dans lequel le composant est chauffé dans une atmosphère de CO et de H₂.

13. Procédé selon l'une quelconque des revendications 10 à 12, dans lequel le composant est chauffé pendant de 3 à 6 heures.

25 14. Procédé selon l'une quelconque des revendications 10 à 13, dans lequel le composant est refroidi dans de l'air.

15. Utilisation d'acier bainitique selon l'une quelconque des revendications 1 à 6 dans des raccords durcis destinés à des tiges de forage durant un forage avec marteau perforateur en haut refroidi par air réalisé au-dessus de la surface du sol.

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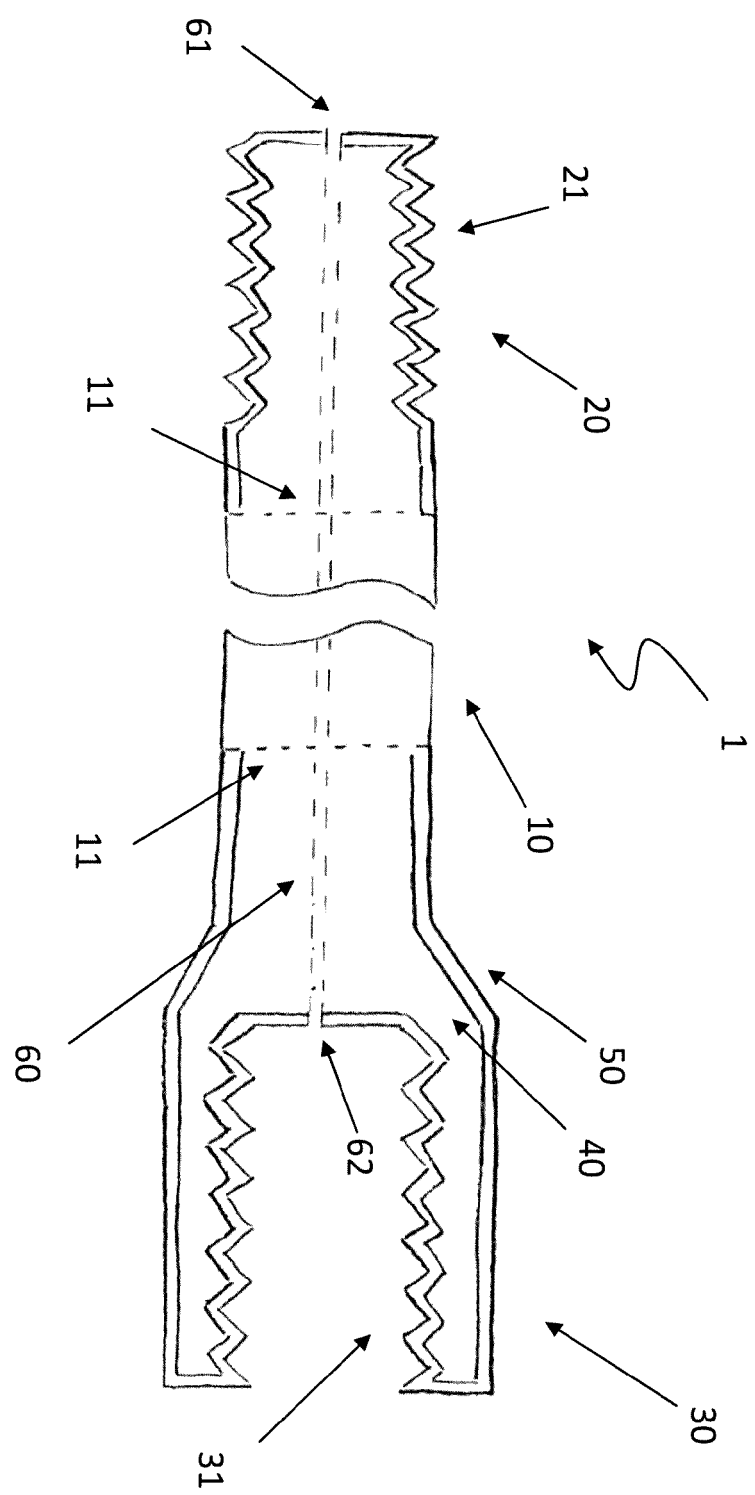


Figure 1

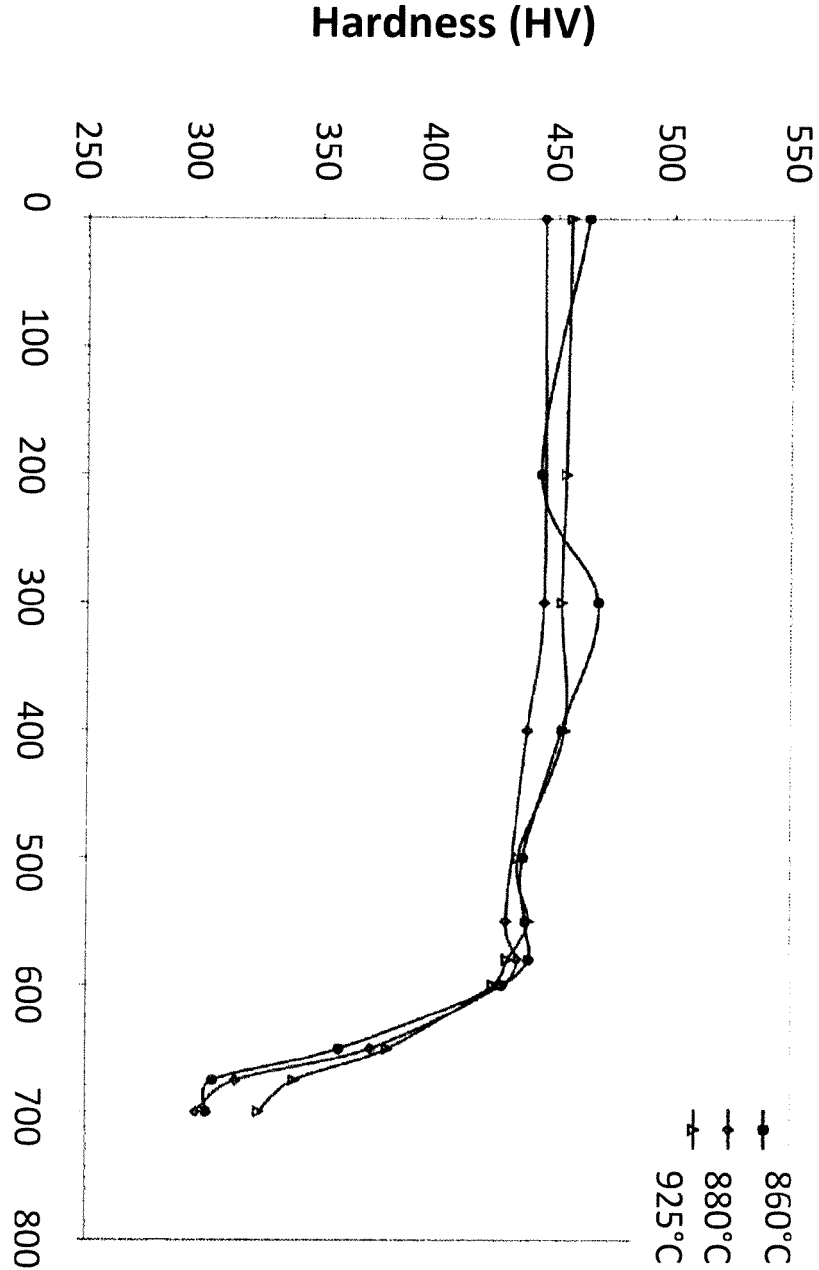


Figure 2

Sample no	Surface (HV1)		Radius(HV1)		Centre (HV1)		Austenitizing Temp	Reheating Temp
4X1	458	470	482	472	461	461	860°C/1h	As quenched
4X2	448	443	474	495	447	451	860°C/1h	As quenched
4X3	418	447	450	470	420	455	860°C/1h	200°C
4X4	446	463	468	479	472	478	860°C/1h	300°C
4X5	432	435	466	466	468	445	860°C/1h	400°C
4X6	417	414	429	448	469	438	860°C/1h	500°C
4X7	412	433	436	465	438	438	860°C/1h	550°C
4X8	435	436	442	432	439	446	860°C/1h	580°C
4X9	428	420	431	421	426	435	860°C/1h	600°C
4X10	360	352	363	357	363	353	860°C/1h	650°C
4x11	297	298	310	307	302	311	860°C/1h	675°C
4X12	301	297	302	302	301	305	860°C/1h	700°C
4X13	428	433	464	478	441	448	880°C/1h	As quenched
4X14	431	443	469	454	418	428	880°C/1h	As quenched
4X15	-	-	-	-	-	-	880°C/1h	200°C
4X16	422	450	464	455	443	434	880°C/1h	300°C
4X17	424	426	449	455	441	432	880°C/1h	400°C
4X18	-	-	-	-	-	-	880°C/1h	500°C
4X19	433	432	436	435	416	420	880°C/1h	550°C
4X20	426	415	431	448	436	445	880°C/1h	580°C
4X21	421	428	425	432	422	436	880°C/1h	600°C
4X22	376	363	384	375	361	370	880°C/1h	650°C
4X23	319	304	318	314	310	319	880°C/1h	675°C
4X24	298	296	297	300	289	303	880°C/1h	700°C
4X25	448	455	466	474	458	462	925°C/20min	As quenched
4X26	443	420	469	468	455	455	925°C/20min	As quenched
4X27	443	441	465	458	463	454	925°C/20min	200°C
4X28	449	443	463	462	449	448	925°C/20min	300°C
4X29	441	426	472	465	462	456	925°C/20min	400°C
4X30	438	436	432	445	436	417	925°C/20min	500°C
4X31	439	436	449	445	436	425	925°C/20min	550°C
4X32	408	441	448	434	427	419	925°C/20min	580°C
4X33	415	410	435	426	415	440	925°C/20min	600°C
4X34	384	380	373	383	377	376	925°C/20min	650°C
4X35	338	335	340	343	337	342	925°C/20min	675°C
4X36	324	325	325	325	328	318	925°C/20min	700°C

Table 4: Results from reheating tests

Figure 3

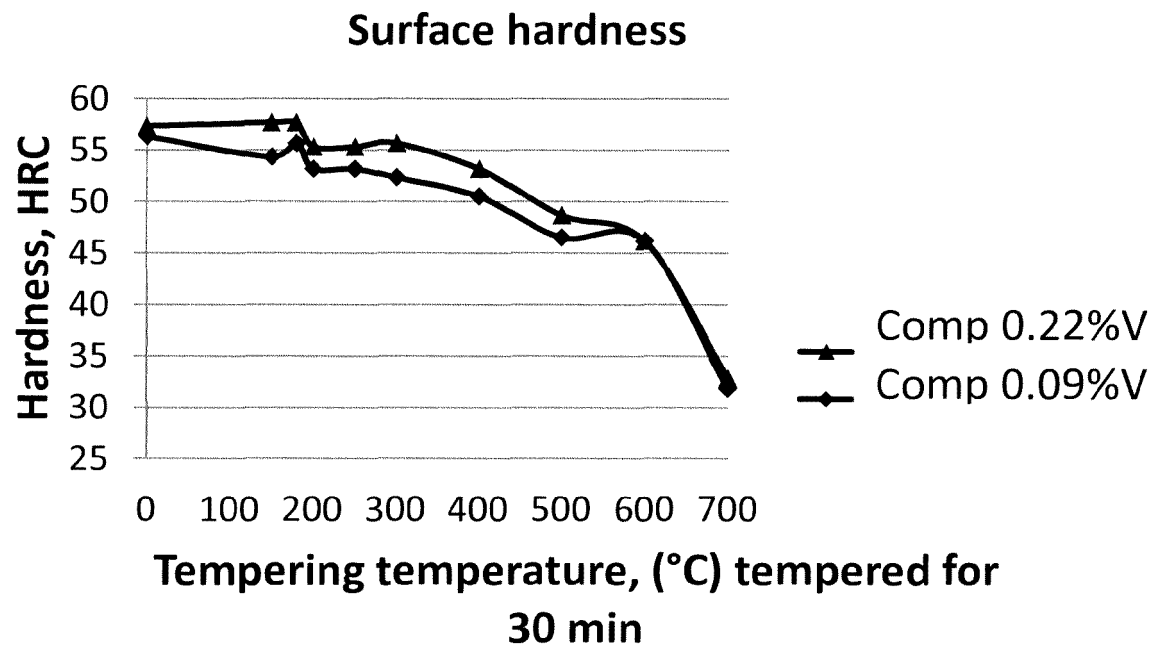


Figure 4

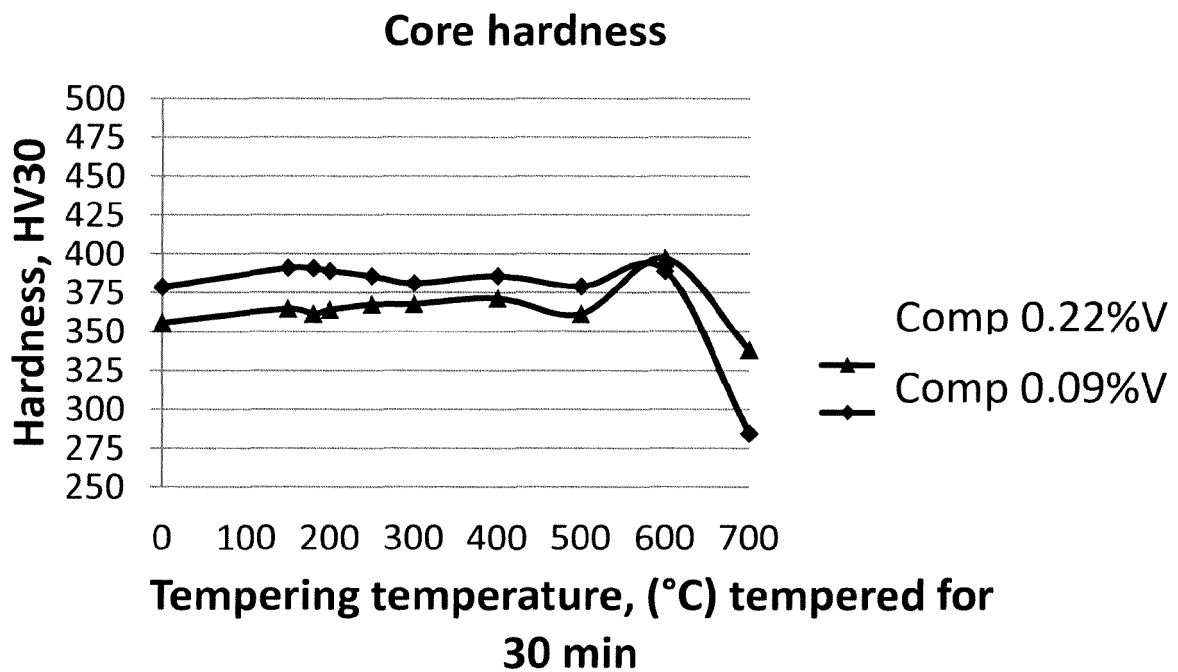


Figure 5

Pressure: 100kPa; 0.019 C; 0.9 Si; 0.75 Mo;
1.2 Cr; 0.2 V; 1.8 Ni; 0.78 Mn; 0.005 N

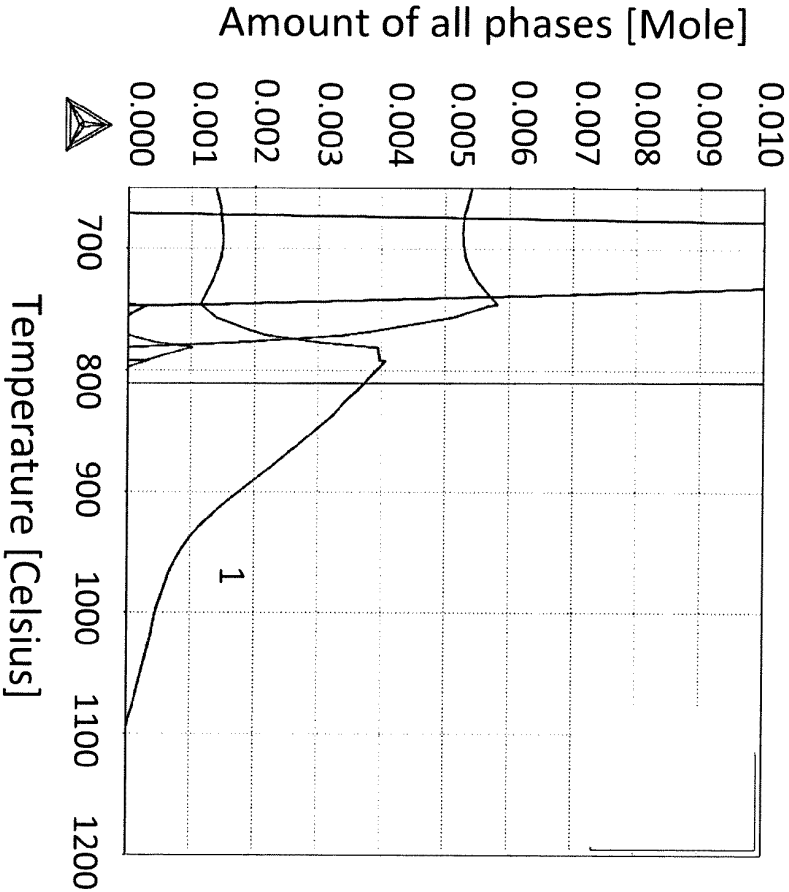


Figure 6

Pressure: 100kPa; 0.019 C; 0.9 Si; 0.75 Mo;
1.2 Cr; 0.2 V; 1.8 Ni; 0.78 Mn; 0.012 N

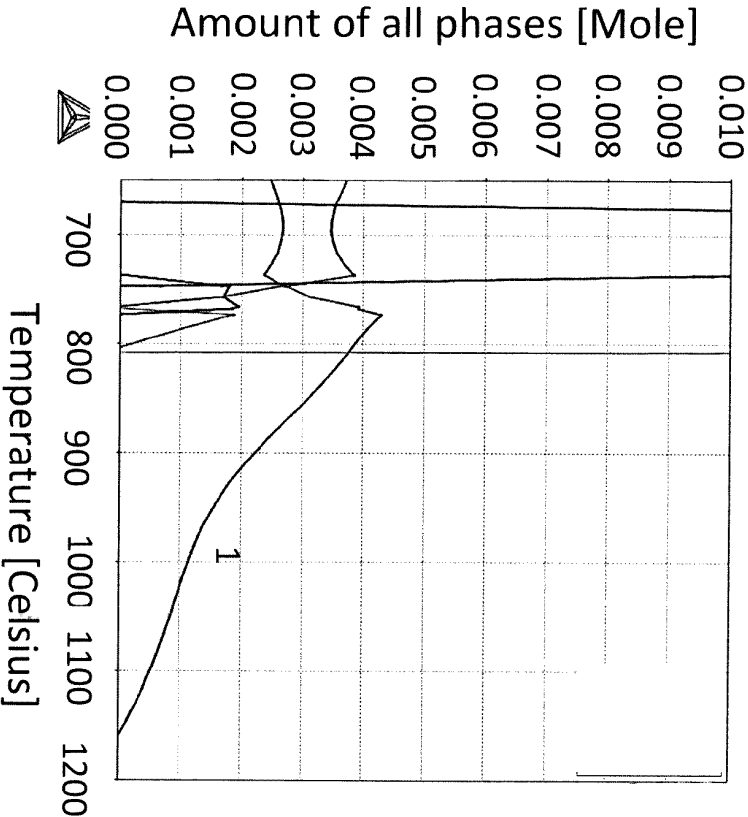


Figure 7

Pressure: 100kPa; 0.019 C; 0.9 Si; 0.75 Mo;
1.2 Cr; 0.3 V; 1.8 Ni; 0.78 Mn; 0.005 N

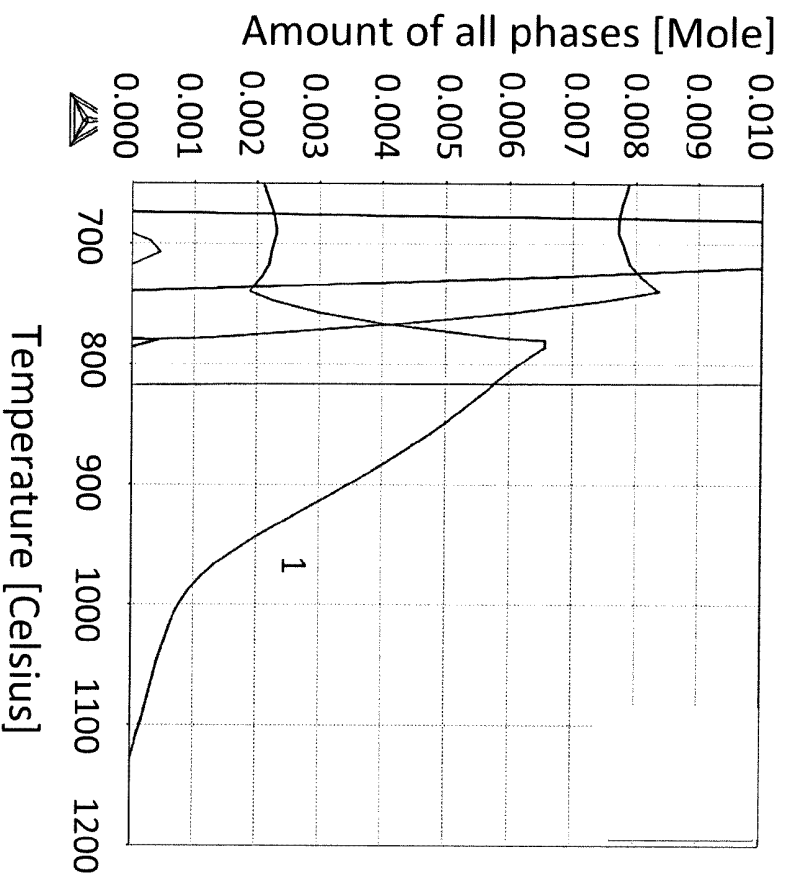


Figure 8

Pressure: 100kPa; 0.019 C; 0.9 Si; 0.75 Mo;
1.2 Cr; 0.3 V; 1.8 Ni; 0.78 Mn; 0.012 N

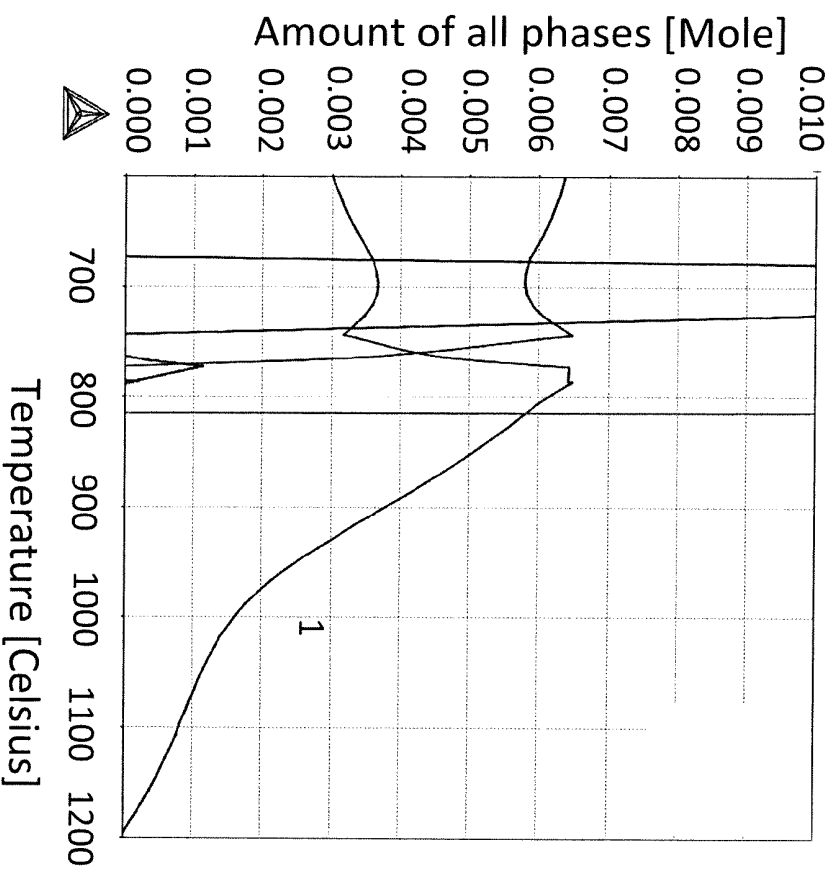


Figure 9

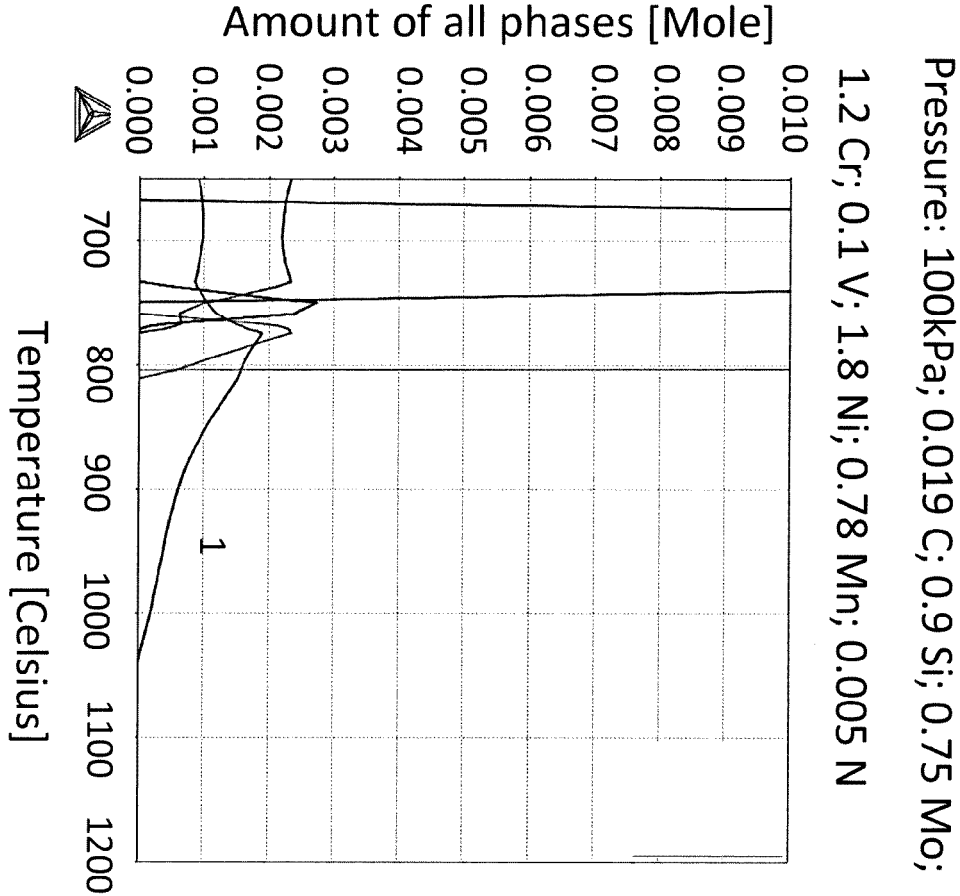


Figure 10

REFERENCES CITED IN THE DESCRIPTION

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