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(54) **METHOD FOR PRODUCING STAINLESS STEEL**

(57) A method of suppressing embrittlement of stainless steel in a stainless steel production process comprising the steps of
• manufacturing stainless steel in a conventional stainless steel manufacturing process

• annealing the stainless steel in an annealing step
• cooling the steel in a cooling step or quenching the steel in a quenching step and
• applying a magnetic field to the stainless steel during the cooling or quenching step

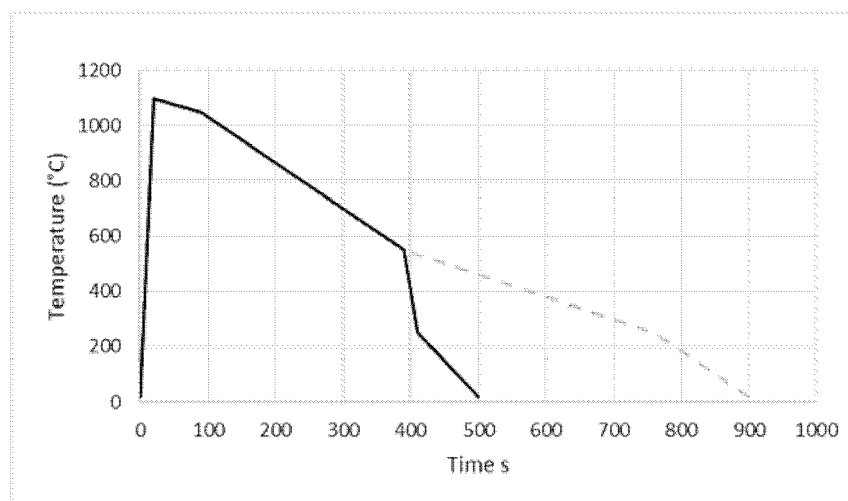


Figure 1

Description

FIELD OF THE INVENTION

- 5 **[0001]** The present invention relates to a method of suppressing embrittlement of steel. Particularly, the method comprises suppressing embrittlement of duplex grades and ferritic grades with an external magnetic field.

BACKGROUND

- 10 **[0002]** The excellent properties of stainless steels are in part owed to the heat treatments performed during the final processing steps. For the stainless steel products to have the required properties, such as good corrosion resistance, formability and strength, for long service life, the heat treatments performed need to be closely controlled. While the heat treatment enhances these desired properties, it also can "sensitize" parts, when not performed adequately. The risk for sensitization generally increases as the stainless steel grades becomes higher alloyed i.e. higher Cr and Mo levels. Resident times in the range of 700°C to 950°C for example, must be avoided to prevent the formation of sigma phase and chromium carbides both of which can reduce the corrosion resistance and mechanical properties of high alloyed austenitic and duplex stainless steel grades. To avoid this, cooling after heat treatment is preferred to be performed by water quenching in water baths or water spraying depending on the product form. For duplex stainless steels another temperature region than should be avoided as much as possible is between 250°C to 500°C. Within this temperature region and phenomena known as spinodal decomposition can occur.

- 20 **[0003]** Spinodal decomposition, often also referred to as 475°C embrittlement, is a degradation of duplex materials which drastically compromises the toughness. For example at 475°C, the decomposition and embrittlement of the ferrite phase, in duplex and ferritic grades, can occur to the extent that the impact toughness can be reduced by 50% after just holding for 3 minutes at temperature. At lower temperatures, 250-350°C, the decomposition still occurs but at a significantly lower rate, up to 10s of thousands of hours at temperature is required for the same level of negative impact on the properties.

- 25 **[0004]** It has recently been realized that for thick gauge parts (such as quarto plates) a certain degree of spinodal decomposition can occur during the cooling/quenching of the part after solution annealing. Cooling/Quenching is most typically performed in batches in water baths (or heavy spraying in-line). But even in baths with high water agitation the cooling rate of the parts can be too slow resulting in undesired material properties. Air-cooled parts that cool slower than quenched parts are even more susceptible to spinodal decomposition as it takes longer time for the material to cool through the critical temperature region. It is well established that highly alloyed duplex grades and ferritics can be susceptible to precipitation of unwanted phases such as sigma phase during cooling, but as mentioned previously the fact that spinodal decomposition and thus can also play a significant role in the reduction of properties during the cooling/quenching step is new finding. Lower alloyed duplex and ferritic grades that do not suffer sigma phase precipitation might still be susceptible to spinodal decomposition when water quenched albeit to a lesser extent. However, some products of lower alloyed grades such as bars can be air-cooled to produce as-rolled products. Since the cooling rate when cooling in air is relatively slow, the grades produced in this manner will be susceptible to spinodal decomposition.

- 30 **[0005]** JPH09217149A shows that 475°C embrittlement can occur during the cooling of thick gauge casts with grains size larger than 50µm. They teach that an advanced cooling schedule is required to suppress sigma phase, 475°C embrittlement and induced stress, including that the cooling rate needs to be higher than 10°C/min from 500°C to 300°C to limit 475°C embrittlement. Such a cooling approach is however, not possible in an in-line quenching process or practical in industrial scale batch annealing using large tanks for quenching.

- 35 **[0006]** Further, 10°C/min is still too slow in some materials, e.g. in thick gauge materials having a grain size most more than 50µm to avoid spinodal decomposition. The highest possible cooling rate, at large plate thickness, due to material properties is still not fast enough to suppress 475°C embrittlement sufficiently.

- 40 **[0007]** JP2006212674A describes how ferritic grades can suffer from brittleness due to 475°C embrittlement in hot rolled coils if not adequately cooled during coiling. A solution is proposed by starting the coiling at a specific temperature such that the tip of the coil enters the mandrel just above the embrittlement temperature range and thereafter is rapidly cooled during the coiling by the mandrel which has an in-built cooling function. This approach however, is not practically possible on plate material or bars for example. Nor, is it possible in an in-line process where for example annealing is directly followed by pickling.

- 45 **[0008]** CN108315549A claims a method for eliminating aging embrittlement of duplex stainless steel, which is subjected to an electric pulse treatment on aged duplex stainless steel, characterized in that the parameter range of the pulse processing is: frequency 1 to 200 Hz, pulse width 20 µs to 1 ms, current 10 to 2000 A, function Time 1 to 6h. This method is said to heal the aged duplex steel after long exposure to the 475°C regime, but does not prevent the occurrence during the processing of the steel.

SUMMARY OF THE INVENTION

[0009] The inventors have recently found that 475°C embrittlement can even occur during the cooling/quenching step. The properties of the materials subjected to 475°C embrittlement during cooling have been determined to be somewhat reduced in terms of impact toughness, albeit not to the same extent as isothermal aging at 475°C as described above. However in combination with the presence of sigma phase (also formed during the same cooling/ quenching) the impact toughness can be lowered further to below acceptable levels, thus limiting plate thickness or quality of the delivered material. Suppression of this embrittlement is therefore vital for achieving high standard products.

[0010] The invention is defined by what is disclosed in the independent claim. Preferable embodiments are set out in the dependent claims.

[0011] It is an aim of the present invention to overcome at least some of the disadvantages of the prior art and provide a method of suppressing embrittlement of stainless steel.

[0012] According to a first aspect of the invention there is provided a method of suppressing embrittlement of stainless steel in a stainless steel production process comprising the steps of manufacturing stainless steel in a conventional stainless steel production process, annealing the stainless steel, cooling/quenching the steel and applying a magnetic field to the steel. The magnetic field is applied to the steel in the cooling or quenching step.

[0013] Considerable benefits are obtained by means of the present invention. By means of the present invention it has surprisingly been found that embrittling of stainless steel is suppressed, and thus high quality stainless steel products may be provided.

[0014] Further features and advantages will become apparent from the following description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0015] The present invention is illustrated in more detail referring to the attached drawings in which:

Figure 1 shows different cooling curves attained by using a GLEEBLE testing equipment. A GLEEBLE equipment is used generally for thermomechanical testing of materials and for simulating physical processes for example industrial heat treatments and cooling thereafter. This equipment is therefore ideal for simulating different cooling conditions that are relevant for the 475°C phenomena. The sample is heated to the desired annealing temperature, 1100°C in this example and then controlled cooled to simulate the cooling conditions typical for industrial production of quarto plates, bars etc. In Figure 1 a typical industrial cooling curve is shown by the dashed line, having an average cooling rate of 60°C/min throughout the whole cooling from 1100°C to under 100°C and a rapid cooling curve shown by the solid line, with normal industrial cooling of 60°C/min to the start of the critical embrittlement temperature range (550°C) followed by rapid cooling at >500°C/min through this range was simulated.

Figure 2 is a plot of SANS data (Intensity $I(Q)$ [cm^{-1}] vs. inverse wavelength Q [nm^{-1}]) showing evidence of separation of phases (increase in peak intensity and shift to lower wavelength at longer exposure times) at 450°C for super duplex grades. This figure also shows how the external magnetic field (1.5T) suppresses the peaks (spinodal decomposition) and therefore embrittlement.

Figure 3 is a plot of SANS data (Intensity $I(Q)$ [cm^{-1}] vs. inverse wavelength Q [nm^{-1}]) showing evidence of separation of phases (increase in peak intensity and shift to lower wavelength at longer exposure times) at 450°C for duplex grades. Figure 3 also shows how the external magnetic field (1.5T) suppresses the peaks and therefore embrittlement.

Figure 4 is a plot SANS data (Intensity $I(Q)$ [cm^{-1}] vs. inverse wavelength Q [nm^{-1}]) showing how the initial normal industrial cooled material at 60°C/min (identified as "slow" in the plot) has an influence on the nanostructure already in the as received condition compared to the more rapid cooling condition, >500°C/min through 550-350°C (identified as "fast" in the plot).

DETAILED DESCRIPTION

[0016] The present invention relates to a method of suppressing embrittlement of stainless steel in a stainless steel production process comprising the steps of manufacturing stainless steel in a conventional stainless steel manufacturing process, annealing stainless steel in an annealing step with an annealing temperature range of 900-1250°C, quenching the steel in a water bath, or in any other suitable quenching media/process, including air-cooling with or without forced air, through the critical temperature range of 350°C to 550°C and to below 350°C, particularly below 200°C, preferably 150 °C or below, suitably 100 °C and applying a magnetic field to the steel during either the annealing step or the cooling/quenching step or during both the annealing step and the cooling/quenching step.

[0017] FIG 1 shows that different cooling rates can influence the properties of the duplex stainless steels with regards to 475°C embrittlement. The normal industrial process cooling (dashed line) gives a low impact toughness strength as some degree of spinodal decomposition already occurs during this cooling time period. In the case where the material is rapidly quenched at 550°C through to 350°C (solid black line) the impact toughness is significantly higher (table 1). This is since the spinodal decomposition has not had time to occur. For air cooled samples, since the resident time in the critical region would be significantly longer than the normal process cooling, the impact toughness would logically be even lower than that for the process cooled sample.

[0018] FIG 2 The curves in Fig 2 represent different heat treatments and process conditions for super duplex material. The material aged for 500 hours at 450°C has a large peak in intensity at around $Q\ 0.007\text{nm}^{-1}$ indicating a relatively large amount of spinodal decomposition and probable embrittlement. After 6 hours at this temperature the peak intensity is less and shifted somewhat to a higher inverse wavelength ($Q\ 0.01\text{nm}^{-1}$) showing that the phase separation is less as could be expected given the shorter exposure time. This trend continues for the sample held at 2 hours with a lesser but still very significant peak. In comparison, the material held at 450°C for 6 hours and 2 hours in the presence of a magnetic field (1.5T) shows no sign of phase separation as no peak intensity is observed from the background level. This shows that in the presence of the magnetic field the 475°C embrittlement is fully suppressed in the isothermally aged condition up to at least 6 hours.

[0019] FIG 3 Show the same findings as for FIG 2 except for duplex grade 2205 rather than super duplex grade 2507. Despite the fact that duplex grades are less sensitive to the embrittlement than super duplex grades on account of the fact that they have less Cr. Fig 3 still shows that at 450°C for 6 hours a significant degree of spinodal decomposition does exist and a somewhat affected nanostructure can be observed even after 30mins. Again, even for this grade in the presence of an external magnetic field the phase separation is significantly suppressed.

[0020] FIG 4 plots the SANS data (Intensity $I(Q)$ [cm^{-1}] vs. inverse wavelength Q [nm^{-1}]) for samples cooled under different conditions. The total exposure time to the 475°C temperature region is less than for the conditions in FIG 2. The general background intensities are different between the "slow" and "fast" cooled sample curves due to the difference in general microstructure e.g. different austenite spacing. But the slight curvature in intensity (data points show a higher intensity than the background reference line added to the plot) at about 0.01nm^{-1} measures a change in the nanostructure and indicates that the slow cooled sample shows an initial phase separation, whereas the fast cooled sample seems to be unaffected. The process cooling rate of the slow cooled sample in FIG 4 was similar to the conditions of the slow cooled samples in FIG 1 (50-70°C/min and Table 1) which also had the low impact toughness. This tells that the production produced material can have some degree of phase separation and embrittlement already in the as received condition. The degree of phase separation and thus embrittlement would be more severe in air-cooled parts when the cooling rate would be much less than 50°C/min. The "fast" cooled sample in FIG 4 was rapidly cooled ($>500^\circ\text{C}/\text{min}$) faster than is possible under normal process quenching conditions. Therefore, in the real process it is not possible to cool thick plates, such as quarto plates, fast enough to avoid some initiation of embrittlement. Application of an external magnetic field overcomes this problem and allows for material to be delivered free from spinodal decomposition/embrittlement.

EMBODIMENTS

[0021] The present invention relates to a method of suppressing embrittlement of stainless steel in a stainless steel production process. In an embodiment the process comprises the steps of providing an annealed stainless steel, cooling or quenching the steel in a cooling or quenching step; and applying a magnetic field to the stainless steel. Cooling/Quenching is most typically performed in batches in water baths (or heavy spraying in-line) or air cooled. The magnetic field is applied to the stainless steel during the cooling or quenching step. It has been found that embodiments of the invention are suitable for all types of stainless steel. Thus, in an another embodiment the step of providing an annealed stainless steel comprises stainless steels produced by all processes such as, but not limited to, melting of raw and / or scrap materials, casting stainless steel into ingots, slabs, blooms or atomized metallic powder or further processing the stainless steel by rolling, pressing or forming into billets, plates, sheets, strips, coils, bars, rods, wires, profiles and shapes, seamless and welded tubes and/or pipes, formed shapes, near net shape powder metallurgy and profiles, thereafter casting either by ingot casting or continuous casting to be subsequently hot rolled and or cold rolled, or forged ready for final annealing in the temperature range of 900°C to 1250°C. In a further embodiment the stainless steel melt can be used to produce metallic powder for isostatic pressing or additive manufacturing for annealing and cooling or quenching.

[0022] In one embodiment raw materials such as nickel and molybdenum are melted together with scrap metal in an electric arc furnace with or without vacuum oxygen decarburization. Electric arc furnaces are particularly efficient for the purposes of embodiments of the present invention.

[0023] In an embodiment the stainless steel is tapped into a mould. Moulds may be made from various materials known to those skilled in the art. In one embodiment the stainless steel is tapped into a copper mould. In a further embodiment the stainless steel is continuously cast into slabs.

[0024] As mentioned above stainless steel manufacturing processes comprise an annealing step. Such processes

may comprise one or more annealing steps in the temperature range of 900°C to 1250°C. In an embodiment the annealing step has the purpose of homogenizing the stainless steel and softening and dissolving secondary phases. Secondary phases include, e.g. carbides, nitrides and intermetallics, like sigma phase. Thus, in an embodiment the annealing step comprises raising the temperature of the steel to a temperature in excess of 900°C. The annealing step

may be carried out in batch furnaces for batch annealing or by in-line annealing, in embodiments.

[0025] Stainless steel manufacturing processes also include a cooling or quenching step. The number of quenching steps is usually equal to the number of annealing steps. In an embodiment the cooling or quenching step reduces the temperature of the steel to maintain the material properties of the steel.

[0026] In an embodiment the quenching step reduces the temperature of the steel from the annealing temperature to a temperature below which secondary phases or embrittlement can no longer form or occur i.e. 350°C or lower, particularly below 200°C, preferably 150 °C or below, suitably 100 °C or below by a quenching method selected from batch quenching in water tanks, in-line quenching, spraying and other suitable quenching techniques as used in the stainless steel industry when producing duplex stainless steels. In some embodiments the quenching step reduces the temperature of the steel to room temperature or to a temperature in range of room temperature to 350°C. For the purposes of this application room temperature means a temperature in the range of 20 to 25°C. Cooling to less than 20°C has no extra benefit.

[0027] In another embodiment a cooling step reduces the temperature of the steel by air-cooling either by forced air or natural cooling to room temperature.

[0028] In a further embodiment the magnetic field has a field strength in the range of >0.2 T, preferably >1.0, most suitably 1.5-3.0T. Increasing the magnetic strength to greater than 3.0T has no extra benefit as the material will have reached magnetic saturation. By means of the magnetic field, embrittling of the stainless steel is suppressed.

[0029] As mentioned above, embodiments of the invention are suitable for all stainless steels. In a particular embodiment the stainless steel is ferrite containing stainless steel or martensite containing stainless steel.

[0030] The following non-limiting examples illustrate at least some embodiments of the invention.

EXAMPLES

[0031] Duplex grades are sensitive to 475°C embrittlement. Impact toughness testing (Charpy impact tests) is a standardized testing method (for example in accordance to ISO 148-1:2016, ISO 17781 and well established as a test method for determining the presence of detrimental phases for duplex grade such as described in ASTM A923 method B) ideally suited for determining if a material has been sensitized in some way, for example by 475°C embrittlement. Impact toughness tests have been performed at -40°C on duplex grades that have been subjected to different cooling conditions from the normal annealing temperature as shown in Figure 1. For this purpose a GLEEBLE instrument was used to simulate a typical cooling curve for thick plates materials when quenched in a water bath whereby the cooling through the critical temperature range 550-350°C was at a normal rate, dashed line in Figure 1. In comparison, other samples were cooled under the same conditions down to 550°C and then rapidly quenched through the critical temperature range to completely eliminate the onset of 475°C embrittlement, solid line in Figure 1. The samples used for this GLEEBLE simulation and impact toughness testing were standard size 55x10x6mm with a v-notch for impact testing. 6mm thickness allowed for precise temperature control throughout the cooling step. The results in table 1 from the cooling conditions in Figure 1, show that with normal process quenching the impact toughness is lower than the samples where rapid quenching was used to avoid embrittlement, showing that 475°C embrittlement exists under the normal cooling condition of thick plates.

[0032] Small-angle neutron scattering (SANS) has been applied to highlight the nanostructure evolution due to the spinodal decomposition of the duplex steels after exposure to temperatures around 475°C either isothermally or during process cooling. Likewise, SANS has been used to show that the spinodal decomposition is suppressed after the same exposures with an applied external magnetic field. Specifically, SANS measures the spinodal decomposition in the nanostructure i.e. on the atomic level, that exists with the development of Fe-rich and Cr-rich (demixing) domains when duplex steels are exposed to temperatures 250-500°C. It is this phase separation that leads to the embrittlement and limits the impact toughness for example. A sample size of 10x10x1mm was used. 1 mm thickness allows for a good signal strength and also avoids multiple scattering that can be otherwise experienced with thicker samples. 10x10mm dimensions are ideal for giving a homogenous temperature across the whole sample in the cases where heated samples are required.

[0033] Using the SANS equipment a number of trials have been performed to highlight the presence of the spinodal decomposition in duplex steels under different conditions, showing how sensitive this material is to this phenomena of 475°C embrittlement and how it can be suppressed in the presence of a magnetic field of for example 1.5T.

[0034] Fig 2 and 3 shows how the phase separation can clearly be detected using SANS for two different austenite-ferrite duplex stainless steels. Materials that have been previously annealed and quenched have been held at 450°C for 6 and 500 hours. The peak intensity increases and the position of the peak moves towards lower Q (nm^{-1}) with increasing time showing that the spinodal decomposition proceeds with time. This is more evident in the Figure 2

compared to Figure 3 due to the higher alloying content, in particular chromium, of the super duplex grade in this Figure 2. Tests have also been performed on samples held at 475°C for up to 6 hours in the presence of an applied external magnetic field of strength 1.5T. These tests showed that the degree of phase separation and thus embrittlement can be suppressed to very low and insignificant levels as indicated by the absence of the intensity peaks otherwise observed without the magnetic field. This applies for both duplex grades in Figure 2 and 3, showing that even when a large amount of spinodal decomposition potentially exists (Figure 2) this can be suppressed in the presence of the magnetic field.

[0035] Fig 4 shows that under normal process quenching which is relatively slow cooled (approximately 60°C/min) as for thick plate material, some phase separation does exist, as indicated by the slight intensity peak in the as-received conditions, compared to the rapidly cooled material. As already mentioned, rapidly cooling (>500°C/min) as fast as the "fast sample" in figure 4 is not possible in many plates, thus the need for a means of suppressing the 475°C embrittlement, such as using a magnetic field, is desired.

[0036] It has been indicated that the kinetics of decomposition in duplex alloys is significantly suppressed, and that embrittlement should also be significantly delayed, in the presence of an applied external magnetic field.

[0037] To this purpose, an external magnetic field of >0.2T should be applied to the stainless steel to be cooled or quenched during the entire cooling or quenching process of the part, preferably applied during cooling or quenching from below 600°C. The magnetic field is applied in such a way that the entire material to be cooled or quenched is encompassed by the magnetic field of >0.2T.

Table 1. Impact toughness results for the different cooling profiles from FIGURE 1.

Process	Impact toughness (J)
Normal process "slow" cooling through 550-350°C temperature range	75
Rapid quench through 550-350°C temperature range	215

Claims

1. A method of suppressing embrittlement of stainless steel in a stainless steel production process comprising the steps of:

- manufacturing stainless steel in a conventional stainless steel manufacturing process;
- annealing the stainless steel in an annealing step;
- cooling the steel in a cooling step or quenching the steel in a quenching step; and
- applying a magnetic field to the stainless steel

characterised in that the magnetic field is applied to the stainless steel during the cooling or quenching step.

2. The method according to claim 1, wherein the stainless steel production process comprises

- melting a raw/scrap material for stainless steel production;
- casting stainless steel into ingots, slabs, blooms or atomized metallic powder;
- further processing the stainless steel by rolling, pressing or forming into billets, plates, sheets, strips, coils, bars, rods, wires, profiles and shapes, seamless and welded tubes and/or pipes, formed shapes, near net shape powder metallurgy and profiles.

3. The method according to claim 1 or 2, wherein the raw material is melted in an electric arc furnace with or without vacuum oxygen decarburization.

4. The method according to any of the preceding claims, wherein the stainless steel is tapped into a mould.

5. The method according to any of the preceding claims, wherein the stainless steel is tapped into a copper mould.

6. The method according to any of the preceding claims, wherein the stainless steel is continuously cast into slabs.

7. The method according to any of the preceding claims, wherein the annealing step comprises raising the temperature of the steel to a temperature in excess of 900°C for homogenization, softening, and dissolving secondary phases.

EP 3 981 889 A1

8. The method according to any of the preceding claims, wherein the cooling or quenching step reduces the temperature to maintain the material properties of the steel.
- 5 9. The method according to any of the preceding claims wherein the annealing step is carried out in batch furnaces for batch annealing or in-line annealing.
- 10 10. The method according to any of the preceding claims wherein the cooling or quenching step reduces the temperature of the steel from the annealing temperature to a temperature below 350°C, particularly below 200°C, preferably 150 °C or below, suitably to room temperature either by a quenching step (batch quenching in water tanks or in-line spraying) or cooling step (as air cooling either by forced air or natural cooling).
- 15 11. The method according to any of the preceding claims, wherein the magnetic field applied during quenching has a field strength in the range of >0.2 T, preferably >1.0T, suitably 1.5T to 3.0T.
- 20 12. The method according to any of the preceding claims, wherein the stainless steel is ferrite containing stainless steel or martensite containing stainless steel.

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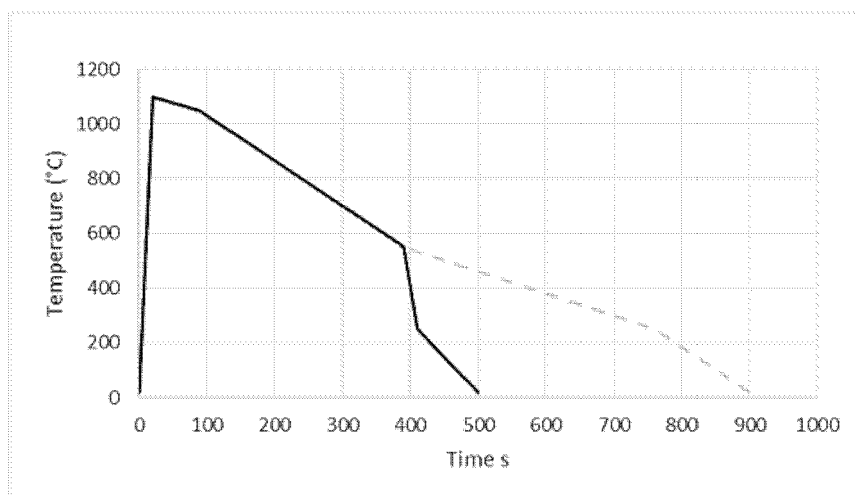


Figure 1

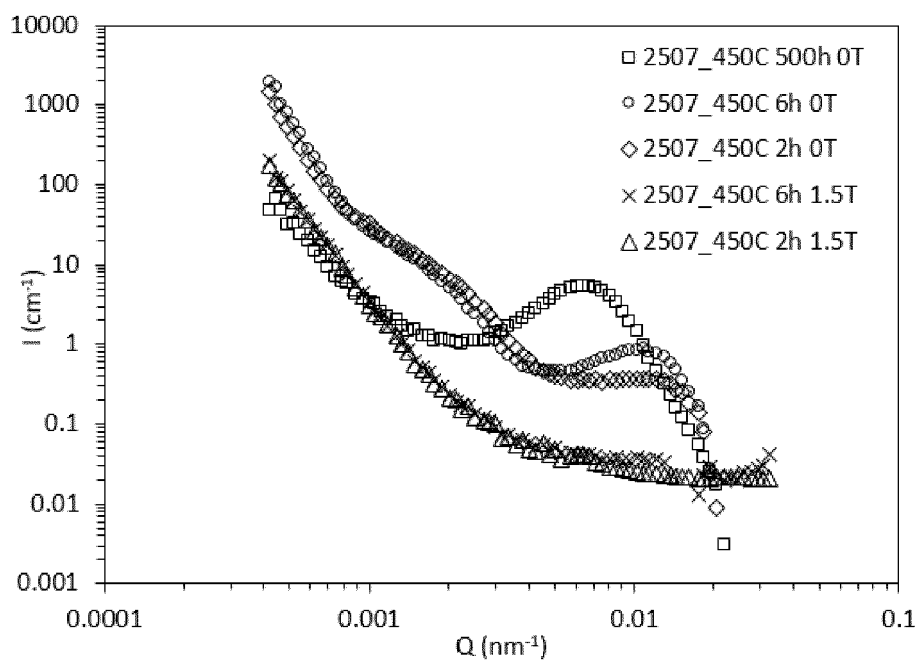


Figure 2

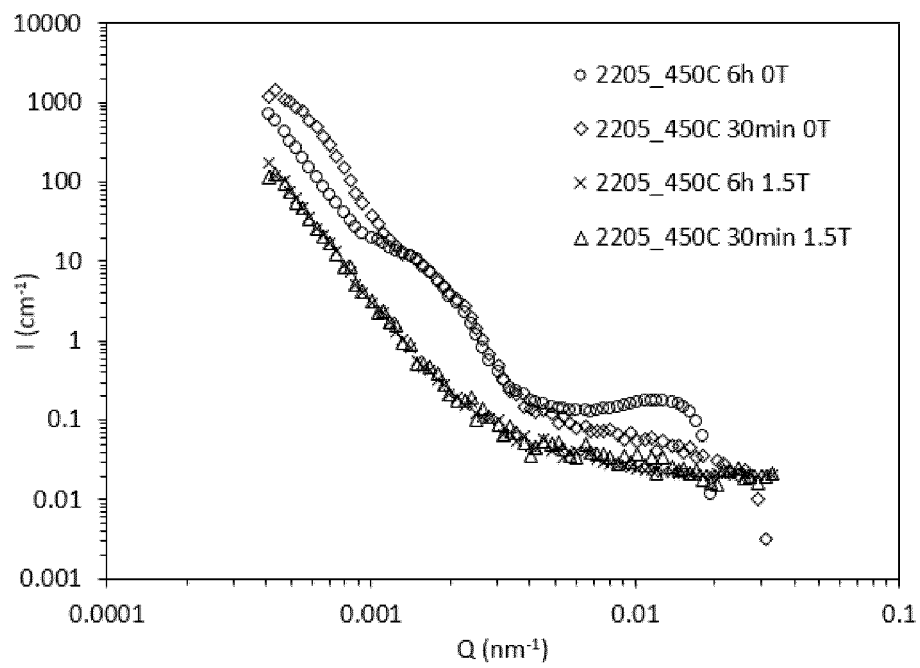


Figure 3

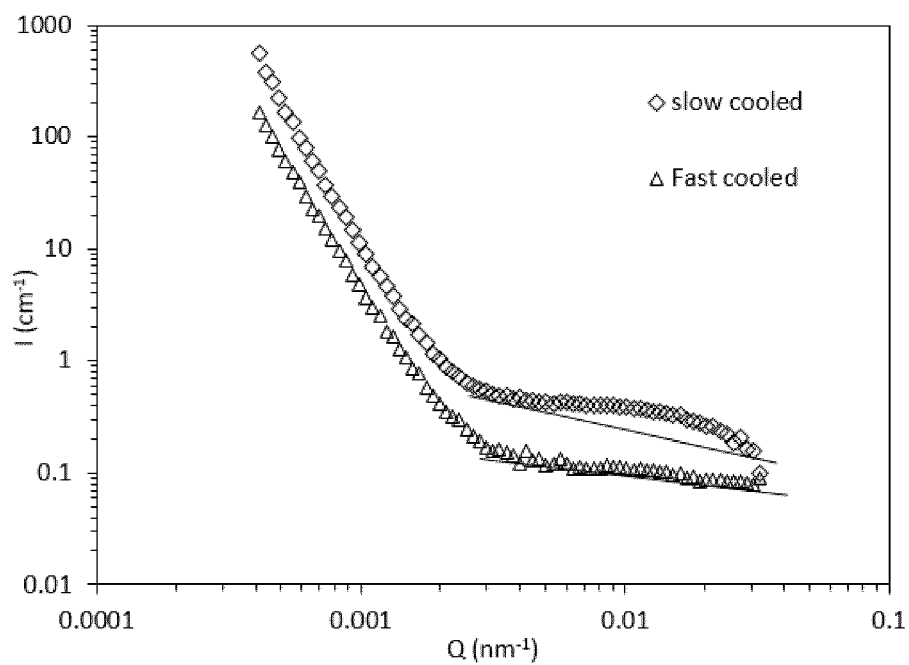


Figure 4



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Application Number
EP 20 20 1101

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The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 17 November 2020	Examiner Chebeleu, Alice
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
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EP 20 20 1101

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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REFERENCES CITED IN THE DESCRIPTION

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