Influence of Mechanical Processing and Heat Treatment on Microstructure Evolution in Nickel Free High Nitrogen Austenitic Stainless Steels

G. BALACHANDRAN, M. L. BHATIA, N. B. BALLAL1) and P. Krishna RAO1)

Defence Metallurgical Research Laboratory, Hyderabad 50058 India. 1) Indian Institute of Technology, Bombay, India.

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A series of nickel free high nitrogen austenitic stainless steels were processed by various mechanical processing and heat treatment conditions to study the evolution of microstructure at every stage of processing. About eleven steels varying in composition, especially in terms of carbon contents, were processed through forging, rolling, solution annealing, cold rolling and warm rolling etc. The microstructure at every stage of processing was studied. The hot rolled condition, hot rolled and cold rolled condition gave deformation induced fragmented grain structure. The high carbon steels in the hot rolled conditions showed banding and grain boundary carbonitride precipitation. There were also intragranular carbide precipitation in some of the high carbon steels. The solution treated microstructure in all steels showed extensive annealing twins and the lattice parameter increased with carbon and nitrogen contents. Cold rolling followed by solution treatment lead to development of slip lines in the fully austenitic matrix due to strain hardening of the matrix. The 600°C warm rolled condition showed grain boundary carbide precipitation.

KEY WORDS: nitrogen austenitic steels; Cr–Mn–C–N steels; mechanical processing; microstructure; heat treatment.

1. Introduction

Modern high technological applications require unique combination of properties such as high strengths with high toughness, non-magnetic behaviour, corrosion resistance etc. Nickel free high nitrogen steels based on Cr and Mn satisfy these requirements. The alloying constituents and processing adopted significantly influences the microstructures and hence ultimately the mechanical properties of these austenitic stainless steels. It is necessary to understand the various microstructures that can be developed in the steel, at different processing condition to attain versatile range of properties. In these steels, there exists a wide variety of microstructures, some of which are extremely detrimental and some of which are beneficial. Hence, understanding the microstructure is crucial to realising the best properties in the steel.

These steels are fully austenitic steels in the solution annealed and subsequently cold rolled condition.1,2) Since, these steels are age hardenable and there are three types of precipitates that are reported that is a grain boundary carbide, lamellar nitrides and intragranular carbides.1) While the intragranular carbides are reported to be beneficial, the other two types of carbides are reported to be detrimental. The precipitation in these steels, occur not only in the aging conditions, but also during certain annealing heat treatments and mechanical processing.1,3) Further, the beneficial intragranular carbides are reported to form in high carbon steels subjected to low temperature aging.1) The precipitation studies during mechanical processing in the above studies are made in steels with lower Mn and Cr contents and in steels with additionally nickel constituent.

In this paper, attempt is made to analyse the microstructure evolution in a series of nickel free high nitrogen austenitic stainless steel compositions with a base composition around 18wt%Cr–18wt%Mn, with significantly varying carbon and nitrogen contents. The effect of mechanical processing and heat treatment conditions on the steel microstructures as a function of steel composition has been examined. The study enables choice of appropriate processing parameters for obtaining a desired microstructure in these steels with significantly varying C and N contents. The influence of carbon on various stages of mechanical processing and microstructure development has been examined.

3. Experimental

A series of steel compositions chosen for this study is given in Table 1. The steels were processed through conventional ESR process. The cast ingots were subject to various thermomechanical processing conditions shown in Fig. 1.

The cast ESR ingots were forged at temperature between 1150 to 1050°C. In the case of the steels M2 and M3, a part of the as-forged ingot was solution treated. In the case
of ingot 2/1, the steel was hot forged by about 95% to a cross section of 30 mm x 30 mm from 150 mm diameter ingot. Resoaking was carried out thrice during forging at 1150°C. All other ingots, of about 100 mm diameter, were hot forged to a thickness reduction of 73% between 1150 to 1050°C. After 4 to 5 passes, the sheets were reheated. The interpass time was maintained at about 1 to 2 min. The 30 mm thick slab was reduced to 6 mm thick sheets corresponding to about 80% reduction. The steels after hot rolling were not kept at high temperature for insufficient time for recrystallisation during hot rolling. The fine and they are partially recrystallised. This may be due to gone deformation. The grain size is non-uniform and very high. It was also subjected to varying temperature and time for studying the grain size variation.

Solution treated steels, M2 and M3 were warm rolled at 600°C to a reduction of 40%. They were soaked for 1 hr at 600°C and subjected to warm rolling. The rolls were at room temperature. Sections of the warm rolled steels were further cold rolled up to the extent of reduction in thickness possible, which were found to be 13% in steel M2 and 7% in steel M3.

The higher carbon hot rolled steels 3/1, M4 and M8 showed banded structures, which is typically shown in Fig. 2(b). The banding is found to be due to lamellar nitride precipitation higher magnification of which is shown in Fig. 2(b). The intensity of banding is observed to be severe in the transverse direction than longitudinal. The results of X-ray diffraction studies were conducted in the various steels using Philipps makes diffractometer with model 1830 generator, using Cu Kα radiation. The X-ray powder pattern of the various samples were analysed for phase constituents and lattice parameters. The lattice parameter of the steels studied have been calculated using Nelson and Riley parameter.4

4. Results and Discussion

4.1. As-hot Deformed Condition

The microstructure in the as-hot deformed condition is shown in Figs. 2(a)–2(c). Extensively deformed grain structure was observed in most steels, especially in low carbon containing steels. The steels were hot rolled between 1150 to 1050°C. After rolling, the steels were allowed to cool to room temperature in the ambient condition. The low carbon steels show deformation induced highly strained grain structure as shown in Fig. 2(a). In some regions, slip lines could be seen within a grain. The microstructures obtained in the low carbon steels in the present evaluation compares very well with those reported by Efimenko et al.51 and Ikegami et al.60 These studies were also for low carbon steels. The compositions used by Ikegami et al.60 had slightly more nickel contents and also additional carbide forming elements. The precipitates in the as-hot deformed condition were not characterised by Ikegami et al.60. The studies by Efimenko does not talk about any precipitation during or after deformation. In both these studies and as well as low carbon steels in the present study, the grain boundaries are found to be highly deformed, annealing twins have undergone deformation. The grain size is non-uniform and very fine and they are partially recrystallised. This may be due to insufficient time for recrystallisation during hot rolling. The steels after hot rolling were not kept at high temperature for recrystallisation but were instead allowed to cool to room temperature. The longitudinal and transverse microstructures are almost uniform. As high nitrogen steels are low stacking fault energy materials,7,8 during hot rolling, it is possible that dynamic recrystallisation is the more predominant mechanism rather than dynamic recovery by which hot working takes place.7

The higher carbon hot rolled steels 3/1, M4 and M8 shows banded structures, which is typically shown in Fig. 2(b). The banding is found to be due to lamellar nitride precipitation higher magnification of which is shown in Fig. 2(b). The intensity of banding is observed to be severe in the transverse direction than longitudinal. The results of X-

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**Table 1. Composition of the steels examined.**

|    | C  | N  | Cr | Mn | Si | S  | O  | P  | Al |
|----|----|----|----|----|----|----|----|----|----|
| M2 | 0.12 | 0.53 | 18.50 | 17.50 | 1.16 | 0.02 | 0.0015 | 0.025 | <0.01 |
| M3 | 0.10 | 0.68 | 17.50 | 16.60 | 0.91 | 0.018 | 0.001 | 0.007 | <0.01 |
| M4 | 0.16 | 0.66 | 19.00 | 10.80 | 0.48 | 0.04 | 0.001 | 0.009 | <0.01 |
| M8 | 0.22 | 0.83 | 19.80 | 18.30 | 0.57 | 0.027 | 0.001 | 0.005 | <0.01 |
| 1/1 | 0.05 | 0.72 | 19.10 | 16.90 | 0.91 | 0.044 | 0.0020 | 0.007 | <0.01 |
| 1/2 | 0.07 | 0.72 | 19.10 | 16.75 | 0.91 | 0.091 | 0.0030 | 0.008 | <0.01 |
| 2/1 | 0.62 | 0.56 | 18.00 | 18.00 | 0.56 | 0.014 | 0.0060 | 0.006 | <0.01 |
| 3/1 | 0.33 | 0.75 | 19.70 | 18.60 | 0.79 | 0.027 | 0.0035 | 0.049 | <0.01 |
| 3/2 | 0.32 | 0.56 | 19.10 | 16.60 | 1.00 | 0.023 | 0.0040 | 0.075 | <0.01 |
| 4/1 | 0.11 | 0.66 | 19.75 | 18.40 | 1.05 | 0.02 | 0.0057 | 0.010 | <0.01 |
| 5/1 | 0.09 | 0.60 | 19.40 | 18.60 | 0.34 | 0.018 | 0.0045 | 0.050 | <0.01 |
| 5/2 | 0.09 | 0.76 | 19.40 | 18.50 | 0.31 | 0.010 | 0.0035 | 0.012 | <0.01 |

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**Fig. 1. Mechanical processing and heat treatment conditions used in the evaluation of the steels.**
Ray diffraction studies in the steels in hot rolled conditions is shown in Table 2(a). The austenite peaks are shown in Table 2(b). These peaks appear in all austenite containing samples. The presence of lamellar nitride was confirmed to be Cr₂N type nitride in steels M4 and M8. Lamellar nitride formation is observed in steels 3/1, M4 and M8 because the steels were cooled to room temperature after hot forging in ambient conditions. The formation of lamellar precipitates is attributed to slow cooling after hot forming. ¹, ², ³ The presence of higher carbon content has promoted formation of such lamellar precipitates. The lamellar nitride precipitate was not observed in the case of low carbon (≈0.1% C) steels processed under similar conditions. It is known that in molten high nitrogen steels, carbon has a positive interaction with nitrogen and the values are strong, involving higher order interaction parameters.⁸ There does not seem to be any published information on interaction of carbon on nitrogen in solid austenite phase. However, similar trend may be presumed in the solid state as well. In that case, there could be a tendency for nitride to precipitate in high carbon steel due to positive interaction between C and N. The lamellar nitrides are reported to preferentially grow on defect prone grain boundaries⁹ and hence they form banded structures in high aspect ratio grains obtained in rolled high carbon steels. In the case of steel 2/1 with 0.62% C content, equiaxed grains are observed with intragranular precipitates. Further, steel M8 which has 0.22% C content showed Cr₂C₆ type carbides in the matrix as shown in Table 2(a) during XRD studies. The formation of carbides and nitrides have been reported during aging of nitrogen austenitic steels at low aging temperatures and in steels with higher carbon contents.¹ Slow cooling in the ambient conditions subsequent to hot rolling could have result in the formation of carbides, in addition to lamellar nitrides as observed in steel M8. Further, the carbides could be also residual carbides formed after hot forging and subsequent cooling, which remained even during hot rolling.

The steel 2/1 alone was evaluated in the as hot-forged
condition. The optical micrograph shows more equiaxed grains in steel 2/1 as shown in Fig. 2(c) compared to the other hot rolled steels. There was no banding like that observed in steels M4 and M8. But very fine intragranular precipitation could be observed. Similar microstructure after forging was reported by Hsiao et al. in a 0.4% C steel. But, that study did not characterise the precipitates formed. The XRD analysis on forged steel 2/1 as shown in Table 2(a) indicate presence of Cr$_2$N type nitrides and absence of carbide peaks. It is surprising that steel 2/1, which had 0.6% C carbon content, did not show carbide precipitates in the matrix. According to Hsiao et al., intragranular type carbide formation occurs in high carbon low nitrogen steels aged at low temperature and longer aging times. The lamellar type Cr$_2$N precipitates are formed at high temperatures in high nitrogen low carbon steels. Steel 2/1 has high carbon (0.6%) and high nitrogen (0.5%) contents and at the lower limits of forging temperatures can form Cr$_2$N nitrides in accordance with literature and not the carbides. However, it may be expected that cooling subsequent to forging, can lead to the formation of carbides in this steel. But, the carbide formation is reported to take place at long aging times at low temperatures after forging and the nitrogen content is also high. Hence, detectable carbide peaks were not observed in the in steel 2/1. Though, the precipitation observed in steel 2/1 is intragranular type and confirmed to be Cr$_2$N, it does not have lamellar morphology similar to that observed in steel M4 (0.16% C) and 3/1 (0.33% C) as observed in Figs. 2(a) and 2(b). Two probable reasons could be assigned for Cr$_2$N appearing as dispersed phase in steel 2/1. The lamellar Cr$_2$N precipitate formed at the lower limits of forging temperature could have undergone precipitate coarsening at that temperature and subsequent cooling. Secondly, the lamellar grain boundary Cr$_2$N could have initially nucleated at the lower limits of forging temperature as lamellar grain boundary precipitates. The precipitates could have got sheared, deformed and partially dissolved during resoaking and forging treatments to show-up as intragranular precipitates.

The lattice parameter of the bulk austenite phase is also shown in Table 2(a). The lattice parameter values in the as hot deformed condition are slightly less than that in the solution treated condition. The precipitation of nitrides could have depleted the matrix from nitrogen, which could have lead to reduction in lattice parameter values.

Thus, in the as-hot deformed condition low carbon steels show partially recrystallised grains, while high carbon steels of the order of 0.2% C, show formation of detrimental lamellar precipitation, banding etc. However, very high carbon contents of the order of 0.6% could be manipulated to give intragranular precipitation of nitrides in the matrix. The low carbon steels with the partially recrystallised grains and total austenite phase could give significant improvement in the strength with adequate ductility. Hence, high carbon steels under this condition give undesirable microstructures.

### 4.2. Hot Rolled + Cold Rolled Condition

The as-hot rolled steels which showed ability for further cold rolling was subjected to 40% thickness reduction. Only steels with low carbon levels could be rolled. The higher carbon steels such as 3/1, M4 and M8 cracked during initial deformation by about 5%. The cracking in high carbon steels may be attributed to the brittle grain boundary lamellar precipitation which remained as banded structures as shown in Fig. 2(b). During cold rolling, the nitride precipitates could nucleate cracks along the precipitate zones due to their brittle nature. The matrix also gets excessively strain hardened because the precipitate hinders the dislocations and cause a highly strained matrix structure, which leads to cracking. The deformed low carbon steels show oriented grains along the direction of rolling as shown in Fig. 3. There are slip lines seen within the highly fragmented and partially recrystallised grain structure. This is indicative of high strain hardening of the matrix. The trans-
verse microstructure in the steel shows extensively etched grain boundaries much more intense than in the longitudinal direction. The overall grain structure in this condition was a high aspect ratio anisotropic grain structure with slip lines within the grain. There were no annealing twins seen. The XRD analysis as shown in Table 3, indicate absence of other types of precipitation or formation of strain induced martensite formation.

Thus, low carbon high nitrogen steel can be further subjected to cold rolling after a hot deformation treatment which results in a highly strained austenite matrix which could eventually strengthen the steel. The high carbon steel cannot be subjected to loading after the hot deformed condition as they tend to crack on deformation at room temperature. Hence, application of high carbon steels after hot rolling in load bearing condition is undesirable.

4.3. Solution Treated Condition

All steels subjected to solution treatment at temperature >1000°C show single phase austenitic microstructure with annealing twins. The minimum solution treatment temperature required for making the steel fully austenitic as a function of steel composition is based on a steel with similar composition reported by Hsiao1) is shown in Table 4. It has been reported that higher the carbon or chromium content in the steels, higher is the solution treatment temperatures required.1) It was found that if the solution treatment temperature is not adequate, there is the presence of residual carbides. This was observed in the analysis of XRD data of steels M2, M3, M4 and M8 solution annealed at 1050°C for 30 min as shown in Table 5. The 6 mm thick steel sheets, M2 and M3 showed residual carbides when solution treated at 1050°C for 30 min. The high carbon steels M4 and M8 showed residual carbides even at solution treatment temperature of 1100°C/30 min. When the solution treatment was raised to 1150°C/60 min, the matrix was totally free from carbide precipitates. There was no nitride peaks observed in any of the steels after solution treatment. Thus, to get carbide free austenitic matrix high solution temperatures of the order of 1150°C are required. However, this may increase the grain size of the steel.

The lattice parameter of these steel in the fully austenitic condition is shown in the Table 5. It is seen that, as the total interstitial contents increases, the lattice parameter of the austenite also increases as shown in Fig. 4. This trend has been reported by other authors.1,2) The increase in the lattice parameter was reported to increases the lattice friction during deformation, consequently increasing the strength of the steel. To find the influence of nitrogen alone on lattice parameter, Fig. 5 was plotted, where in the data are plotted for all steels with about 0.1% C. The effect of substitutional solid solution on lattice expansion was not as significant as that of the interstitial contents. Comparison of Figs. 4 and 5, show that carbon has similar influence as nitrogen in expanding the austenite lattice.

The microstructure of all the ESR cast steels, after hot rolling and hot forging followed by solution treatment revealed fully recrystallised austenitic matrix with annealing twins. Typical microstructures of steel M2 and M3 solution treated after forging and rolling is shown in Figs. 6(a)–6(c). The steel M2 and M3 in Figs. 6(a) and 6(b) were hot forged and solution treated at 1100°C/30 min. It is seen that at the same processing conditions of forging and solution treatment, the steel M2 exhibits finer recrystallised grains than steel M3. This is attributed to the presence of some characteristic inclusions. Steel M2 showed presence of AlN inclu-

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| Steel | Lattice parameter of austenite | Other phases/ characteristic plane |
|------|-------------------------------|-----------------------------------|
| M2   | 3.625                         | nil                               |
| M3   | 3.621                         | nil                               |
| 1/1  | 3.619                         | nil                               |
| 5/2  | 3.627                         | nil                               |

Table 4. Minimum solution treatment temperature required for complete austenite based on Hsiao1) on a steel close to 18%Mn–18%Cr base composition.

| Steel | T (°C) |
|-------|--------|
| M2    | 955    |
| M3    | 887    |
| M4    | 985    |
| M8    | 1040   |
| 1/1   | 857    |
| 2/1   | 1246   |
| 3/1   | 1099   |
| 3/2   | 1096   |
| 4/1   | 857    |
| 5/1   | 857    |
| 5/2   | 857    |

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Table 5. Lattice parameter of austenite in the solution treated samples.

| Steel | a0 Å’ | Other Phases with planes |
|-------|-------|--------------------------|
| M2    | 3.624 | Cr23C6 (400),(200),(611)  |
| M3    | 3.630 | Cr23C6 (420),(611)        |
| M4    | 3.636 | Cr23C6 (420),(611)        |
| M8    | 3.634 | Cr23C6 (400),(611)        |
| At higher solution treatment temperatures and fully austenitic matrix |
| M2    | 3.626 |
| M3    | 3.625 |
| M4    | 3.630 |
| M8    | 3.636 |
| 1/1   | 3.632 |
| 1/2   | 3.629 |
| 3/1   | 3.641 |
| 3/2   | 3.631 |
| 5/1   | 3.636 |
| 5/2   | 3.638 |

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Fig. 4. Influence of interstitial % (C+N) on lattice parameter.

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The formation of AlN inclusion in steel M2 was due to the presence of residual Al content about 0.08%. During the primary steel making in induction melting route, aluminium was used as a deoxidant. This deoxidation was needed so that the surface active O levels in the steel can be considerably decreased for better nitrogen alloying. The residual Al, when it was about 0.05 % as observed in steel M2 formed AlN precipitate scavenging some nitrogen from the base steel. The presence of a small quantity of AlN in these steel could be beneficial, as they form inclusions as shown in the EPMA microstructure in Figs. 7(a)–7(b).
which may help in restricting grain growth during plastic deformation. Such effects are reported in conventional Al killed steels.\textsuperscript{10,11} Typical AlN precipitates with needle morphology found as inclusions in the present study is shown in EPMA X-ray map analysis as shown in Fig. 8. This micrograph shows one needle AlN in elongated shape and the other circular as a cross section of the needle precipitate. Steels M2 and M3, both with $\sim 0.1\%$ C content, processed at identical conditions showed finer grain sizes in M2 than M3 as shown in Fig. 6. This is probably because of presence of AlN inclusion in steel M2 than in M3. It has been further reported that these precipitates can dissolve at high temperatures but precipitate during hot deformation. Comparison between solution treated samples after as-forged and as-rolled conditions could be examined as shown in Fig. 6. The hot forged and solution treated steel shows coarser grain size, when compared to rolled and solution treated condition. This may be attributed to the differences in degrees of deformations. The hot forged material had undergone 73\% reduction from a cast ingot while the hot rolled material has undergone 80\% reduction from a prior forged condition. Hence, the extent of deformation endured in the hot rolled condition is greater than the hot forged condition, which probably lead to greater twin density and finer grain sizes.

Two types of samples were used in the solution treatment studies and both the cases there was austenitic matrix with annealing twins. In the first case, solution treatment was carried out in the as hot rolled samples. The solution treated steels subsequent to hot rolling exhibit extensive annealing twins in their microstructure as shown in Fig. 7. In higher carbon steels, the lamellar nitrides and carbides formed during the as hot-rolled condition remained needed to be dissolved and hence higher solution treatment temperature and time is required. In the second case, steels were recrystallised by solution treatment in samples cold rolled by 40\% after prior solution treatment at 1150\degree C. In the latter case, the samples prior to solution treatment did not have any precipitates to start with but had a strain hardened austenite matrix. The microstructure of typical steels recrystallised and subjected to grain growth after cold rolling is shown in Figs. 9 and 10. The samples show completely austenitic grains with annealing twins. The slip steps formed during cold rolling and the grain anisotropy associated with rolling are eliminated and complete recrystallisation has taken place even at 1000\degree C. There is absence of carbides and nitrides at this lowest temperature. These steels were earlier subjected to 1150\degree C solution treatment before cold rolling hence all precipitation formed during hot rolled condition was eliminated. In addition, at 1000\degree C these steels do not age to form nitrides or carbides even in steel with 0.33\% C. The grain size measurements were carried out ignoring the twin boundaries within a grain.

Since steel 2/1 was processed in bar form, the samples used for studying the effect of solution treatment on grain size was carried out in the as-forged samples. Higher solution treatment temperature was applied because steel 2/1
had higher carbon content and nitride precipitates. Further, the samples used were round bars of 12 mm thick. Hence, higher solution treatment temperature of 1200°C for longer time was applied. The grain growth in steel 2/1 is shown in micrographs in Fig. 10 as a function of time and temperature. The grain growth data was analysed to find whether interstitial constituents influence the grain growth in these samples. It was found that the interstitial content in the steel did not have any effect on the grain growth as shown in Fig. 11.

Since the solution treatment conditions adopted in the present study varied both temperature and time to get higher grain sizes, the effect of grain growth could not be directly plotted with time. However, the present solution treatment time and temperature data could be fitted in the Larson and Miller equation,

\[ P = (T+460)(\log(t)+20) \times 10^3 \]

where \( T \) is temperature and \( t \) is time. Using the above equation, the \( P \) value could be initially calculated for the experimental solution treatment time and temperature. From this calculated \( P \) value, the time for a chosen temperature of 1100°C, could be recalculated. This time is termed as the normalised time. The grain growth as a function of this normalised time was plotted against the grain size. The data shows that the grain growth follows the parabolic equation as shown in Fig. 12. This type of dependence is reported in literature. The present analysis shows that the grain growth does not have any specific relation to the interstitial C and N contents in the steel.

### 4.4. Solution Treatment + Cold Rolled Condition

The microstructure of the steels in the cold rolled condition after solution treatment show extensive slip band formation. The slip steps could be observed within the grains as shown in Figs. 13(a)–13(b). Generally, the grains are elongated towards the rolling direction. The low carbon steel as in Fig. 13(a) show comparatively more equiaxed structure compared to the high aspect ratio grain observed in high carbon steel in Fig. 13(b), under the same 40% reduction in thickness during cold rolling. The presence of annealing twins are still observed along with slip steps in the low carbon steel at the same degree of deformation. In high carbon steel, the grains are anisotropic and there is high degree of slip steps seen. Further, the strain hardening associated with the high carbon steel may be higher than that with low carbon steel resulting in greater strengthening in case of high carbon steel.

The steels in the cold deformed condition was analysed for phase constituents in this condition and the results are shown in Table 6. It is observed in all steels that at 40% cold rolling no strain induced martensite was observed in the matrix. It has been reported that both high N and C contents decrease the critical martensite formation temperature at a particular strain and the compositions range of steels in the present study does not lead to formation of strain induced martensite. The analysis of lattice parameters by XRD shows that the lattice parameter value slightly vary from those calculated in the solution annealed condition. Most of the steels show a decrease in lattice parameters. According to Baret et al., plastic deformation is effective in producing stacking faults in low stacking fault energy fcc solid solutions. The deformation faults in fcc metal could produce a shift in certain of certain peaks in a powder diffraction pattern leading to lower lattice parameters.

Thus, high carbon steels show highly strained anisotropic microstructure than low carbon steel. The microstructures of the high carbon steels in this condition appears favourable for use in the cold deformed condition after solution treatment.

### 4.5. Warm Rolled Condition

The steels were warm rolled to see if the prediction by Dulis\(^3\) that the deformation in the warm rolling regime would favour formation of beneficial intragranular carbide precipitation that may eventually improve the mechanical properties. The study by Dulis involved only tensile deformation at warm rolling conditions at very low strain rates in tensile mode tests and no actual thermomechanical processing at warm rolling temperatures. It is difficult to reproduce such a condition in a conventional hot rolling mill. In the present study, the steel was aged at 600°C for 1 hr before commencement of rolling. The rolls were at room temperature and not heated. This temperature was chosen for rolling because below this temperature aging was very re-
tarded\textsuperscript{1} and above 700°C, there could be simultaneous formation of Cr\textsubscript{2}N as precipitation products.\textsuperscript{1}

Only two samples, low carbon steels M2 and M3, with \(~0.1\%\) C were warm rolled at 600°C. The microstructure of the samples after warm rolling and cooling to room temperature is shown in Fig. 14. Extensive grain spalling is observed in steel M3. There is no extensive spalling of the grains from the matrix in steel M2. The reason that grain spalling took place could be due to the weakening of the grain boundary associated with intergranular carbide film precipitation shown in Fig. 15. The microstructure in the grain interior shows slip lines indicative of the steel matrix getting strain hardened along with the carbide precipitation. The spalling is more on steel M3 and this could be attributed to aging associated with higher carbon and nitrogen contents in steel M3 than in M2. It has been reported that higher C and N contents enhance more grain boundary precipitation.\textsuperscript{1} The XRD analysis conducted on warm rolled samples are shown in Table 7 and the precipitate in the matrix was confirmed to be Cr\textsubscript{23}C\textsubscript{6} carbide type.

The formation of a film type carbide, as shown in Fig. 15 could have occurred during initial thermal treatment of the sample at 600°C for 1 hr. Under these conditions grain boundary carbide precipitation could have initiated. Such carbide precipitation, has been reported by Hsiao \textit{et al.}\textsuperscript{1} during aging. The desired intragranular type of precipitates as reported by Dulis\textsuperscript{3} could not be observed in these steels in the 600°C warm rolled condition. Since, even in low carbon steels, grain boundary carbide formation and spalling of grains are observed, in high carbon steels such precipitates cannot be avoided. Hence, other samples were not processed in this condition. Thus, instead of getting the desired intragranular carbide type precipitation, the material was rendered brittle due to grain boundary carbide precipitation and associated grain spalling. Thus, warm rolling conditions does not give intragranular precipitates reported

Table 6. XRD analysis on solution treated and (40\%) cold rolled steel samples.

| Steel | Austenite Lattice parameter (Å) | Other phases |
|-------|--------------------------------|--------------|
| M2    | 3.628                          | Nil          |
| M4    | 3.644                          | Nil          |
| M6    | 3.643                          | Nil          |
| 1/1   | 3.625                          | Nil          |
| 1/2   | 3.622                          | Nil          |
| 3/1   | 3.625                          | Nil          |
| 4/1   | 3.629                          | Nil          |
| 5/1   | 3.624                          | Nil          |
| 5/2   | 3.626                          | Nil          |
by Dulis. 3) Probably controlled temperature rolling equipment could lead to the formation of intragranular type of carbide precipitates.

The warm rolled steels were further subjected to cold rolling to reduce thickness by 13% in steel M2 and 7% in steel M3, to see the effect of cold rolling on the microstructure and cracking tendency. The sheets could be rolled without crack up to this reduction. Beyond this reduction, the steels cracked. The sheets could be rolled to 13% and 12% without cracking. The microstructure of steel M3 is shown in Fig. 15. The steel shows grains getting elongated in the rolling direction, but they still have a thick grain boundary film precipitates along the grain boundary formed in the warm rolled condition. There are slip lines seen within the grain matrix showing that the matrix is hardened. The film type carbide is similar to sensitised conventional stainless steel. The film of carbide is not completely broken during cold deformation. These brittle zones could lead to intergranular failures and hence poor ductility and toughness to the steel.

5. Conclusions

The microstructure evolution during mechanical processing and heat treatment conditions on nickel free high nitrogen austenitic stainless steels with significantly varying carbon contents was examined. The steel in the as-hot formed condition showed partially recrystallised grain structure favourable for high strength development with good ductility. The high carbon steels developed detrimental lamellar precipitation in this condition and appeared as banding. Low carbon steels could be further cold rolled and the microstructure condition showed extensive slip lines generated within the partially recrystallised grain structure. High carbon steels cracked when subjected to further cold rolling. All the steels, inclusive of high carbon steels in the solution treated condition showed complete austenitic matrix with annealing twins. Higher carbon steels when solution treated at lower solution treatment temperature lead to formation of residual carbides. The grain growth in these steels is found to be not a function of the interstitial contents. The lattice parameter was found to increase with carbon and nitrogen in solution. In the solution treated + cold rolled condition, low carbon steels show more equiaxed structure with twins prevailing while the high carbon steels show extensively deformed anisotropic grains. Warm rolling at 600°C in low carbon steels showed formation of weak Cr23C6 type grain boundary film type carbide precipitates, which were brittle and lead to spalling of the grains.

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