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Origin of low temperature toughness in a 12Cr-10Ni martensitic precipitation hardenable stainless steel

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ABSTRACT

Though ductile to brittle transition (DBT) is a typical feature of body centered cubic materials, the present 12Cr-10Ni precipitation hardened martensitic stainless steel exhibited excellent low temperature impact toughness. This was, however, dependant on the aging temperature. 250 °C aging led to higher toughness both at room temperature and at −196 °C (77 K). Specimens aged at 400 and 500 °C, on the other hand, displayed significantly lower sub-zero impact properties. Though martensite packet size, and size distribution, were identical between the two ageing treatments; there was clear evidence of second phase coarsening: from very fine precipitates of less than ~ 10 nm to relatively coarser second phase of ~ 5–25 nm range. It is suggested that precipitate coarsening and associated loss of coherency are the limiting factors to the DBT performance of this important class of material.

1. Introduction

Precipitation hardenable (PH) stainless steels or maraging stainless steels offer high to ultrahigh strength and toughness, corrosion resistance and fabricability. The development of these steels, in the 1940s, was an initiation to the development of corrosion resistant maraging steels, which was further motivated by the shortage and rise in the cost of cobalt in late 1970s [1]. These steels were able to successfully fill the technological gap between the corrosion resistant and tough austenitic stainless steels with lower strength, and the martensitic stainless steels with higher strength but lower in toughness. Though maraging stainless steels have a range of niche applications [2,3] the area of interest pertaining to the present study are applications such as highly stressed aerospace components.

The alloying characteristics of PH stainless steels are manipulated by a precise balance of alloying additions, which decide the mechanical and physical properties. The mechanical properties of these steels depend on the prior austenite grain size, martensite packet size distribution, presence of retained austenite and nature and distribution of precipitates [2,4]. The nature and distribution of the second phase are determined by the chemical composition and the ageing treatment. The heat treatment condition is also an important factor which determines the resistance to stress corrosion cracking (SCC) with peak strength levels having an adverse effect on the SCC resistance. Majority of these alloys retain small amounts of retained austenite which necessitates sub-zero treatment for complete conversion to martensite after solution treatment [1,2]. In addition to this, austenite reversion can also occur during aging, especially when aging is carried out at higher temperatures (> 500 °C). Both retained and reverted austenite are reported to reduce strength and improve impact toughness at room and low temperatures [2,5].

The precipitation process in PH stainless steels is very complex and uncertainties exist on the precipitation sequence and their shape. The precipitates nucleate from solute rich clusters, intermediate precursor phases or on dislocations [4]. The extremely small size of the precipitates (~ 2–5 nm) and the martensitic matrix limits the resolution achievable in transmission electron microscope (TEM). Published literature on TEM studies pertains to samples overaged to 60–100 h, so as to increase the precipitate size [6–11]. However, state of the art characterisation techniques such as three dimensional atom probe, small angle x-ray scattering and small angle neutron scattering have provided good insights into the precipitation hardening processes of several such steels [11–13].

The most widely used alloys among the stainless maraging steels include 17-4 PH, 15-5 PH and 13-8 PH steels. In addition to these, large numbers of similar alloys have been developed for specific aerospace, medical and tooling industries. “Custom 455” and “Custom 465” steels developed by Carpenter Technology Corporation, “1RK91” steel

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Table 1
Chemical composition of the steel used in the present study.

Element	Specification	Achieved
Carbon	0.03 (max)	0.02
Chromium	11.5–2.5	12.04
Nickel	9.0–10.3	9.84
Molybdenum	0.5–0.8	0.73
Titanium	0.15–0.25	0.24
Aluminium	0.2 (max)	0.04
Manganese	0.15 (max)	0.01
Silicon	0.15 (max)	0.03
Sulphur	0.01 (max)	0.003
Phosphorous	0.01 (max)	0.003
Hydrogen	5 ppm	0.5
Oxygen	30 ppm	28
Nitrogen	100 ppm	54
Iron	Balance	Balance

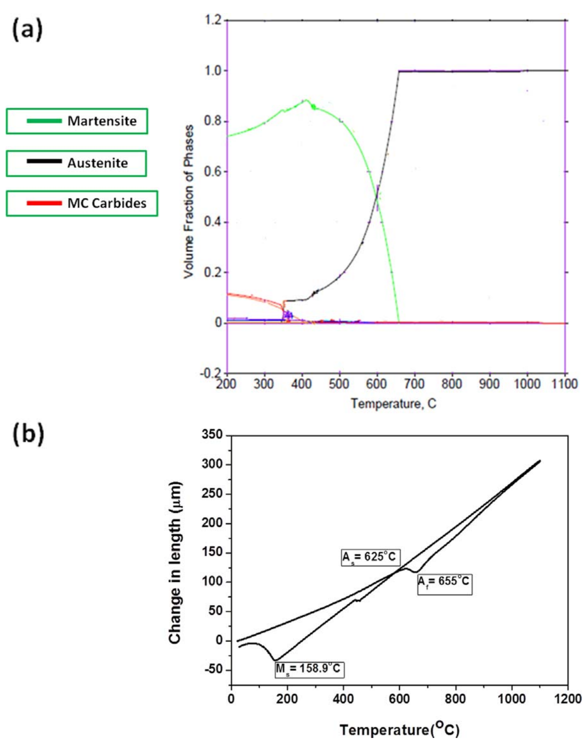

Fig. 1. **(a):** Phase variation chart for 12Cr-10Ni steel calculated by Thermo-Cal™ software in the temperature range of 200–1100 °C. **(b)**: Results of dilatometry for 12Cr-10Ni steel from room temperature to 1100 °C at a heating and cooling rate of 5 °C/min.

developed by Sandvik AB, Sweden are certain prominent proprietary PH stainless steels. 17-4 PH and 15-5 PH steels are strengthened by nano clusters of copper atoms, whereas 13-8 PH steels have NiAl intermetallic precipitates as the hardening phase [2,11]. "Custom 465" steel is strengthened by Ni_3Ti precipitates and "Custom 455" and "1RK91" steel derive strength from copper nanoclusters as well as Ni_3Ti and $Ni_3(Ti, Al)$ precipitates [4]. The precipitation hardened stainless steels, both commercially available and non-standard grades, have been studied by several researchers with focus on the precipitation hardening process [14–20]. Wilson et al. [1] in his review on the developments and applications of maraging steels have discussed on the metallurgy of all classes of maraging steels including stainless maraging steels.

Materials for cryogenic applications require retention of excellent toughness at cryogenic temperatures. Ductile to brittle transition phenomena is the major factor which limits the application of commonly used structural materials (especially high strength steels) for structural applications at cryogenic temperatures. Austenitic stainless steels due to excellent toughness at lowest temperatures, moderate strength levels

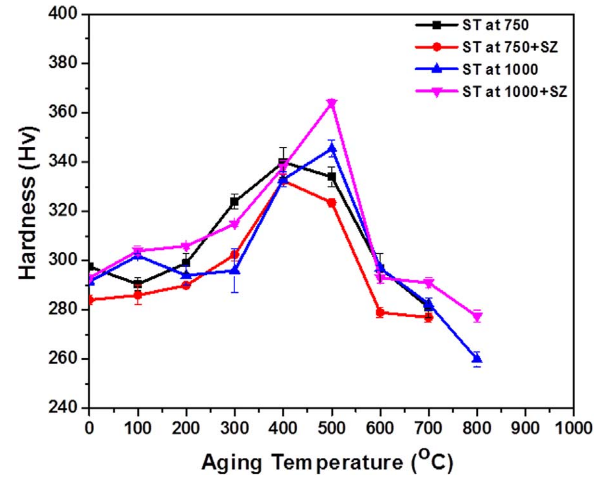

Fig. 2. Isochronal aging curves for 12Cr-10Ni samples solution treated at 750° and 1000 °C, and aged at various temperatures from 100 to 800 °C for 2 h. It is to be noted that both sets of solution treated samples are also subjected to sub-zero (SZ) treatment or quenching in liquid nitrogen. However, SZ samples do not show significant difference in ageing behaviour. Error bars represent standard deviations from multiple measurements.

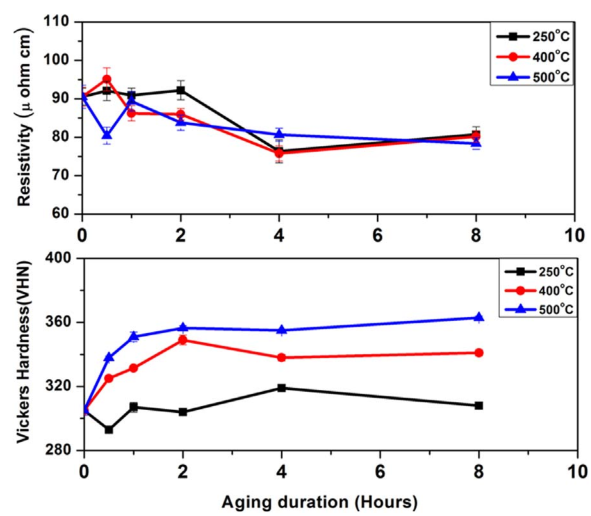

Fig. 3. Variation of electrical resistivity and Vickers hardness with ageing time. These are shown for samples solution treated at 1000 °C and then subjected to three different aging temperatures. Error bars represent standard deviations from multiple measurements.

and excellent fabricability and corrosion resistance, are the most widely used structural material for cryogenic applications. However, low yield strength (~ 250 MPa) is a major drawback for this material, especially for weight critical applications [21–23].

12Cr-10Ni stainless steel (Fe-12Cr-10Ni-0.6Mo-0.25Ti) is an alloy which possesses a good combination of strength and toughness and precipitation hardened by Ni_3Ti precipitates formed during aging following solution treatment. This material is used for structural applications at cryogenic temperatures upto −196 °C (77 K). In order to realise the full potential of this material for structural applications at low temperatures down to 77 K, it is essential to study the mechanical property variations with aging parameters with a view to optimise the heat treatment cycle. The present study focuses on explaining the unusually high cryogenic toughness displayed by this steel and on optimising the heat treatment cycle so as to obtain best combination of strength and toughness at −196 °C (77 K).

2. Experimental

The material used for the present study was processed by vacuum