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INVESTIGATION OF PRECIPITATION IN VANADIUM HSLA STEELS

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Abstract

The precipitation of vanadium carbides in two HSLA steels of compositions, Fe-0.12% C-0.48% V-0.7% Mn and Fe-0.12% C-0.48% V-2% Mn has been studied. The steels were rolled, swaged and cold drawn into 3 mm diameter-wires. Following homogenization treatment at 1200 C and solution treatment at 1250 C the wires were directly quenched and isothermally treated in a Tin bath at two different temperatures of 500 C and 600 C for periods of times up to 60 minutes followed by water quenching. The influence of carbide precipitation after different heat treatments on the hardness and electrical resistivity (at 77 K) has been followed. The changes of properties have been correlated to the microstructural changes investigated by light and electron microscopy. A little change in the resistivity due to precipitation during isothermal treatment has been found in the low Mn steel. The increase in Mn content resulted into retardation of the precipitation process which took place at the interface of the moving austenite/ferrite transformation front and on dislocations in the super-saturated ferrite phase, as depicted from the hardness and resistivity measurements and the electron microscopical investigation.

Introduction

In the past three decades the metallurgy of microalloyed steels has been developed considerably. The high strength to weight ratio of the high strength low alloy steels HSLA, their better weldability and low temperature toughness resulted into spreading of their use replacing a wide spectrum of plain carbon and even some alloy steel applications.

The production of microalloyed HSLA steels combines the use of microalloying elements such as Ti, Nb and V which inhibit recrystallization, with controlled rolling to obtain a deformed fine grained austenite microstructure on completion of rolling.

The ability to model transformation behaviour and predict the final steel microstructure and properties, hence helping in the design of alloy composition and production technology, requires a detailed knowledge of mechanism and kinetics of transformations of steel.

The precipitation of carbides, nitrides and carbonitrides of the microalloying elements of vanadium, titanium and niobium plays a decisive role in the control of mechanical properties through the grain refinement during thermo mechanical treatment which contributes to about 20 - 30 % of the yield strength and the precipitation hardening effect, which contributes to about 10 to 30% of it [1].

The aim of this work is to study the precipitation of vanadium carbides in two V-HSLA steels of stoichiometric carbon to vanadium ratio and having different levels of manganese additions during isothermal treatment at temperature 500 C and 600 C and to explore the effect of treatment temperature and Mn additions on the precipitation strengthening potential. The precipitation process was follow using resistometric, hardness, light and electron microscopical techniques.

Experimental Techniques and Procedures

Two vacuum melted steels of compositions shown below supplied in the form of hot rolled 32 mm square bars were cut into 15 mm diam rods before swageing and cold drawing into 3 mm diam wires.

	C	Mn	V	Si	P	S	N	0
(a)	0.13	0.70	0.47	0.01	0.004	0.003	0.0025	0.004
(b)	0.12	2.06	0.48	0.01	0.004	0.005	0.0040	0.003

The specimens were analysed using Berkin Elmer atomic absorption unit, model 238.

After encapsulation in evacuated sealed quartz tubes the specimens were homogenized for 5 hours at 1200°C and air cooled. They were then solution treated for 15 min at 1250°C and quenched in molten Sn bath and isothermally treated at temperatures 500°C and 600°C for different time intervals between 0 – 1 hr. After this time lapse the quartz tube was broken immediately quenching the specimen into water.

The electrical resistivity of aged specimens were measured at liquid nitrogen temperature (77K) using the four terminal method. Resistance was measured by passing a constant current of 0,5 A from a TC-602 CR North Hills power supply through the specimen in series with a standard resistance of 1 ohm held at constant temperature of 21°C. Potential drop was measured with a 6040 S integrirendes digital voltmeter.

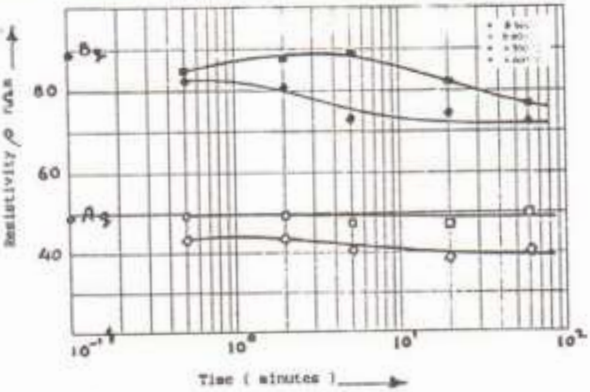
The hardness of the heat-treated specimens was measured using Vickers hardness testing technique. The specimens were prepared using standard methods for light metallographic investigation.

Several techniques were employed to reveal the microstructure details such as bright field, differential interference "Nomarski" contrast using metallurgical microscope type Neophot 21.

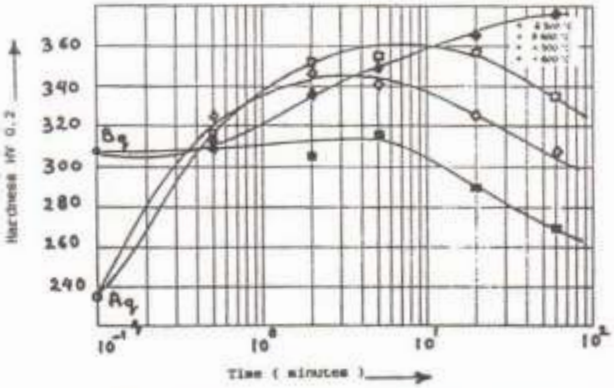
After cutting the specimens with diamond disc, they were mechanically thinned to 0.2 mm and trepanned into 3 mm diam discs. They were then prepared for transmission electron microscope investigations using electrolytic jet polishing technique with a TENUPOL 2 unit. The prepared thin foils were examined using "Jeol 100 CX" transmission electron microscope operating at 100 KV.

Results and Discussions

The precipitation of vanadium carbides, nitrides and carbonitrides in HSLA steels is playing an important role in defining the final mechanical properties through its influence on grain size control during hot processing as well as the possible precipitation



Fig(1): Resistivity vs Isothermal Treatment Time for Alloys (A): (0.12%C,0.46%V,0.7%Mn) and (B):(0.12%C,0.46%V,2.07%Mn) at Temperatures ;500 C and 600 C .



Fig(2): Change of Hardness vs Isothermal Treatment Time for Alloys (A) and (B) at Temperatures 500 C and 600 C .

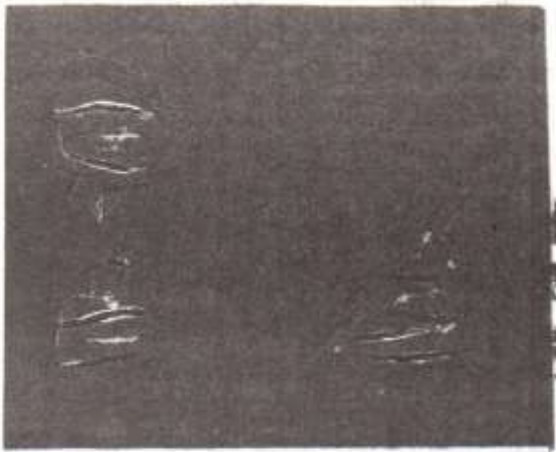
Fig(3) x500
Alloy(A),V-Steel of 0.7%Mn as quenched ,showing large zone of grain boundary ferrite and a martensite/bainite matrix .

Fig(4) x500
Alloy(b),V-Steel of 2.07% Mn , as quenched , showing smaller zone of grain boundary ferrite and fine martensite matrix .

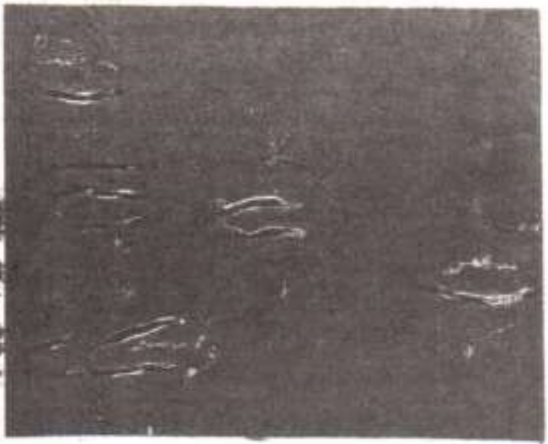
strengthening effect during and after rolling [1,2]. Precipitation in V-steel during isothermal treatment has been previously studied at temperature range between 650°C was explored in the present study using different techniques of electric resistometry, hardness, and both light and electron microscopy. The change of electric resistivity with time for steels (A) and (B) having the base composition of(Fe-0.12% C-0.46%V) but with Mn additions of 0.7% and 2.07% respectively and isothermally treated at 500°C and 600°C for periods of time up to 60 minutes is shown in Fig. (1). The resistivity of both steels shows general tendency to drop with ageing time. This decrease in resistivity can be attributed to the depletion of solute atoms from the primary phase by clustering or precipitation of carbides [6].

The increase of temperature will naturally result into increasing the rate of diffusion and hence precipitation leading to faster drop of resistivity at 600 C as seen in Fig. (1) for both alloys. An abnormal increase in resistivity of 5 nΩm in the alloy (B) of higher Mn aged to 500 C has been observed, a phenomenon which has not been fully understood yet. Similar effect has been previously reported in many age hardening alloys treated at high solution treatment temperature and low ageing temperature including plain carbon steels [7, 8] and HSLA steels [5]. The resistivity increase effect increase effect has been attributed to short or long range order, clustering or zone formation, clustering followed by ordering or electron configuration effects [7, 8, 9, 10, 11, 12]. Although he application of electric resistivity indicated the general tendency of the precipitation rates, such technique could not identify the point of completion of alloy carbide precipitation since reduction in resistivity in the final stages of ageing is due to concurrent alloy and iron carbide precipitation [5], this is confirmed by TEM observation of coarse cementite precipitates in alloy (B) treated for 20 min at 500 C, shown in Fig. (8).

The precipitation hardening effect due to isothermal treatment of alloys (A) and (B) at 500 C and 600 C is shown in Fig. (2). The increase in hardness of the alloys in the as quenched condition from 240 HV in alloy (B) is attributed to the solid solution hardening due to



Fig(5) x200
Alloy(A),Isothermally transformed for 10 min at 500 C,ferrite matrix & small pearlite islands



Fig(6) x500
Alloy(B),Isothermally transformed for 10 min at 500 C , dual ferrite /martensite matrix .

Fig(7) x50K
TEM of Alloy (A) , Isothermally treated for 20 min at 500 C , fiberous VC precipitates .

Fig(8) x20K
TEM of Alloy(B) , Isothermally treated for 20 min at 500 C , coarse carbide precipitates .

increasing Mn content from 0.7% to 2.0%. Typical precipitation hardening curves were found for the alloy (A) of lower Mn at both temperatures of 500 C and 600C, where the hardness increased to reach maxima and softened afterwards for longer ageing times. A maximum hardness gain for this alloy (A) was attained by treatment at 500 C for 10 minutes. The maximum hardness was associated with the precipitation of VC of fiberous morphology of about 15 nm thickness and interparticle spacing of 50 nm as illustrated in the TEM micrograph Fig. (7). The hardening curve of the alloy (B) illustrated rather different behavior. While reaching maximum hardness of 375 HV after 60 min when treated at 600°C, the treatment at 500 C showed no age hardening effect where the harness remained almost unchanged up to 10 min and started to soften gradually afterwards.

It has been found in literature that three situations can arise during direct transformation of microalloyed steels [13];

i) Precipitation at γ/α interface, this mode was recognized by Davenport & Honeycombe (14) who identified two different morphologies i.e. interphase and fiberous precipitates.

ii) Precipitation from super saturated ferrite when held longer at the transformation within the grains, on dislocations and structural imperfections [3].

iii) At low temperature, the ferrite phase may remain supersaturated and carbides may only precipitate on subsequent reheating in ferrite range. All situations may be obtained during heat treatment.

The behaviour of alloy (B) of higher Mn during treatment at 500C indicates that this temperature is too low to activate the precipitation reaching as mentioned in the third situation above due to the effect of Mn in depressing γ/α transformation temperature as indicated by Lq et al. [15]. The addition of Mn causes retardation of γ/α transformation as shown form light micrographs figs (3, 4), of the quenched alloys where grainboundary ferrite zone is smaller in alloy (B) of higher Mn content . Moreover,after isothermal transformation treatment at 500C of alloy (A) for 10 min the microstructure was almost ferritic with small islands of pearlite, Fig. (5), while the alloy (B) of higher Mn subjected to the same treatment showed a mixed bainite/martensite structure, Fig. (6). The precipitated alloy and iron carbides of relatively coarser aggregates in bainite structure left less free carbon to combine with V to form VC which may explain the lack of hardening effect in a that temperature. The growth of cementite precipitates in bainite caused softening after further ageing at 500 C as depicted from TEM micrograph Fig. (8).

Conclusions

- The application of electric resistivity to study the precipitation of VC in V-HSLA steels indicated the general tendency of precipitation rates, however such technique could not identify the point of completion of alloy carbide precipitation.

- An abnormal increase in resistivity of 5 n Ωm in the alloy of 2.07% Mn aged at 500 C has been observed, a phenomenon which require further investigation to be understood.

- The precipitation of VC during isothermal treatment takes place during γ/α transformation from the resulting supersaturated ferrite phase.

- The maximum hardness of the alloys is associated with the precipitation of fiberous VC precipitates of thickness around 10 nm and interparticle distance of 50 nm.

- The effect of increasing Mn addition was to promote the formation of bainite and martensite structures in resulting into increasing the hardness due to solid solution strengthening in contrast to the almost ferritic structure of the alloy with lower Mn content.

- Increasing Mn content to 2.07% also resulted into retarding the

γ/α transformation and depressing the transformation temperature. This effect while delaying the precipitation reaction of the alloy treated at 600 C, it led to the suppression of precipitation at 500C which was too low to activate VC precipitation from bainite/martensite structure less saturated with carbon due to precipitation of coarser carbide aggregates.

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