

## **Forge and heat-treatments in micro-alloyed steels**

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**Abstract :** Improved designs, mostly for light weight component manufacturer have been improvement of forging designs and heat-treatment techniques. Low temperature precipitation strengthening and resistance to austenite grain size coarsening at reheat temperature for forging have been property improvement technique in these microalloyed steels. Studies of peak strain and flow stress at 1123-1423 K have showed increase in peak strain, peak stress and increment in mean flow stress in austenite phases in presence of vanadium. Partial vanadium alloying (1part V substitute for 2 parts Mo) by substituting molybdenum has improved hardenability properties of conventional steels. Ultrafine grained steels have showed strain hardening effects from severe deformation by equal channel angular pressing (ECAP) followed by annealing. The strain induced precipitation of nano-metric sizes have pinned dislocations for strain hardening. Estimation of remaining life for reactor components have been done by simulated experiments under similar conditions as the service exposure. Vanadium in ferritic stainless steel has showed competitive performance, e.g. chloride environment. This has showed equivalent effects like nickel. In welding of microalloyed steel inter-critical grain coarsened heat affected zone (IC GC HAZ) has martensite austenite (M-A) blisters to yield poorest toughness.

**Keywords :** Vanadium, Forging, Steels, Micro-alloy, Hot working, Thermo-mechanical treatment, Heat treatment, Microstructure, Tensile properties, Tempering processes, Vanadium nitride, NDT, DTBT, Ferrite, Embrittlement, Crackling, Martensite, HAZ, Composition, Thermal exposure.

### **INTRODUCTION**

Principles of microalloying to improve mechanical properties has involved following objectives: (a) Low carbon has improved toughness and weldability, (b) grain refinement to improve toughness and increase in yield strength, (c) Precipitation strengthening, (d) Solid solution strengthening and (e) Improved secondary strengthening. Thermo-mechanical forming of bar steels has been basis of forging steel technology. Improved properties in forged steel components have been achieved by additions of microalloy elements, e.g. vanadium and niobium. In forging and post forging process, studies have made to understand dissolution process of microalloying elements. Niobium has been found to increase bending fatigue endurance limit of carburized components by control over austenite grain size, e.g. forged gear. Vanadium has produced intergranular ferrite in medium carbon steel. This has improved the impact properties. Microalloying elements have been precipitate formers, as for example Ti has formed precipitates at high temperature; Vanadium has formed precipitate at relatively lower temperature. Therefore reheat temperature during forging and rolling has redissolved precipitates those have formed at lower temperature where high temperature precipitates remain as such to arrest the austenite grain coarsening rate. Warm forging of automotive components has appeared to be beneficial at 1043 K. The balance among mechanical properties has been observed at this temperature with enough warm ductility. Niobium and vanadium microalloyed steel response at high

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temperature has been determined from constant strain rate, isothermal compression tests on axially symmetrical samples to a strain of unity. Strip rolling has been simulated by four-stage, constant true strain rate compression of the specimen. Interrupted tests have been done to study interrupted loading. Observations have made about the ability of microalloying components to retard to dynamic restoration process. Vanadium has influenced hardening process by narrowing temperature range for crystallization in air-hardening steels. In an ultrafine grained steel, nano-sized isothermal precipitation of vanadium during equal channel angular processing (ECAP) followed by annealing has been more effective to interact with dislocation in ultrafine ferrite grains. Tempering has been softening carbon steel. The softening temperature has appeared to be higher (less than 623 K) in case of microalloyed steels. This is mostly because of carbide forming alloy elements in steel. Higher temperature tempering about 1023 K in 9% Cr-1% Mo-0.2% V steel have showed discontinuous changes in hardness and lattice strain. This has been dissolution of  $M_{23}C_6$  carbides, which has slowed down the growth rate than Ostwald ripening. High temperature impact tests have experienced to simulate radiation embrittlement effects in reactors. Weibull modulus and curves have used to delineate the shift in DBTT by radioactive embrittlement, when plasticity of steel has stopped crack tip blunting mechanism of crack prevention. Tempered martensite embrittlement (TME) has appeared in tempering process of as quenched state from retained austenite decomposition and particle presence within grains. The fracture mode has been transformed from intergranular to transgranular. In welding, heat affected zone (HAZ) has been traditionally accepted as low tough zone. Present context of contemporary developments have referred to inter-critically grain coarsened heat affected zone (IC GC HAZ) which has lowest toughness.

#### Micro Alloyed Forging Steels<sup>[1]</sup>

Optimization for specific application has produced forged microalloyed steels. Requirements of improved properties have been to produce light weight components, e.g. components of automotive engines. Forged microalloyed components have been crankshafts, connecting rods, suspension systems, spindles, driveline components, etc. Forged microalloyed steel components have been produced as direct cooled, i.e. controlled cooled from the forging temperature or as post-heat-treated ones for specific compositions. The heat-treatments have been induction hardening or carburizing. Microstructure and properties have been enhanced in many cases by controlled thermo-mechanical processing. Microalloying of as heat-treated and forged components have been traditional strategy to improve properties. The alloying elements of interest have been vanadium, niobium, chromium, molybdenum, etc. Selection of alloying elements depends on application, related property selection, processing selection and carbon content involved. As for example vanadium improves property by precipitation strengthening to reduce softening tendency during tempering, niobium improves grain refining tendency or control over transformation kinetics, e.g. reducing austenite grain growth and improving bending fatigue strength, increased carburizing temperature and productivity for forged gear applications. Appropriate forging process has appeared to improved strength, machinability and increase in toughness by microalloying vanadium in automotive rear spindle.

Microalloyed bars and forgings have improved properties by precipitation strengthening and balance between strength and toughness is based on carbon content. During processing operation this has influenced dissolution and precipitation processes. Steel forgings may be low carbon

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steels, high carbon martensitic steels, as for example surface hardened gears and medium carbon steels used for springs and shafts etc. Reheating to process by forging, rolling or heat-treating has been redissolution of precipitates and further reprecipitation. Behaviour of precipitate redissolution based on solubility and further reprecipitation below transition temperature after treatment have appeared to vary depending on compound involved from microalloy elements. These precipitating compounds have been NbC, V(C, N) or TiN. (a) Titanium nitride (TiN) has been formed at austenitizing temperature. These have been stable at greater temperature and resist coarsening. TiN in steel has been detrimental to machinability and toughness properties. (b) Vanadium carbonitride V(C, N) has been formed at lower temperature. These have been unstable at austenitizing temperature and dissolve during austenitizing and precipitate at lower temperature. (c) Niobium has been strengthened by solid solubility or fine precipitate formation. This has intermediary performance to those of other two types of precipitates discussed.

Forge reheat temperature has varied as per carbon content. An intermediate to high reheat temperature is required for low carbon content whereas low reheat temperature is required for high carbon steel.

High reheat temperature for low carbon levels have more niobium solubility. Therefore both niobium and vanadium are used for precipitate strengthening. Niobium has been an austenite conditioning element, has suppressed the austenite recrystallization by precipitating preferentially on the deformed substructure formed during thermo-mechanical processing step. Therefore niobium has added potential in as-rolled or as-forged applications. Substantial microstructural refinement has enhanced properties by warm thermomechanical treatment as well.

Low reheat temperature has reduced redissolution of precipitates which has been formed at high temperature. Therefore vanadium has been more substantial than niobium for higher carbon; low reheat consequences of forging, where fatigue strength has been basic requirement. In other words vanadium microalloying has been predominant in these steels. Therefore nitrogen addition has been useful in high carbon; low reheat steels for subsequent precipitation strengthening by vanadium carbonitride precipitates.

As forged microalloyed steel require heat treatment for (a) austenite refinement, (b) precipitation strengthening and (c) tempered microstructure. Titanium and niobium have formed high temperature precipitates. In heat treatment these precipitates have remained undissolved and have suppressed austenite grain growth. This has produced secondary hardening. Vanadium precipitates have been beneficial for carburized specimens or lower austenitizing temperatures, where nitrogen addition has been important.

In case of bar and forging steels, studies have been done on the interactions between chemical composition and processing and their influences on microstructure and properties those control performance. Strategies of microalloying have aimed to apply as forged components with improved properties and with reduction of heat treatment steps.

Vanadium has been treated as primary microalloy element for low carbon sheet and plate products. Pearlites with vanadium precipitate hardening effects have appeared to replace ferrite from this microstructure. Boron has been applied in microalloyed steel to improve hardenability properties.

This is to maintain fine grain sizes by microalloying effects, e.g. AlN during carburizing treatment. Conventional carburizing treatment has been done at low temperature to minimize austenite grain coarsening. Increasing temperature of operation, as for example carburized gears, plasma carburizing has been applied where aluminum nitride (AlN) precipitation has pin down the austenite grain boundaries to reduce coarsening rate during carburizing at relatively greater temperature. Betterment of performance in these directions of sense has been found to be more acceptable for niobium than titanium that is in case of elevated temperature performance. Bar and forged products prone to austenite grain growth during reheat temperature for rolling and forging as well as carburizing temperature has been found to be reduced by niobium fine precipitation more effectively<sup>[1]</sup>.

Universally bar steels produced by following processing steps, (a) melting scrap steel in electric arc furnace, (b) ladle refining, (c) continuous casting of blooms and billets and (d) hot rolling to final bar diameter. Table 1 has showed some applications those have produced from complex shaping of these bars from necessarily at high temperatures. Change to complex shapes have referred to low temperature carbides those form during cooling after changes in shape. Therefore forgings requiring complex changes in shape have referred to vanadium carbonitride precipitation for fine precipitation strengthening effect in steel.

Table 1 : Applications for microalloyed forging steels<sup>[2]</sup>.

Sl.No.	Name of components	Sl.No.	Name of components
1.	Crank shafts	9	Piston shafts
2.	Connecting rods	10	Axle shafts
3.	U-bolts for leaf springs	11	Suspension arms
4.	Steering Knuckle Supports	12	Transmission shafts
5.	Antis way bars	13	Wheel hubs
6.	Induction hardened bars	14	Steering arms
7.	Drive couplings	15	Axle beams
8.	Fasteners	16	Pipe fittings

As forged vanadium bearing steel components has competed highly tempered heat-treated steels. This has been precipitation strengthening effects from vanadium carbonitride during

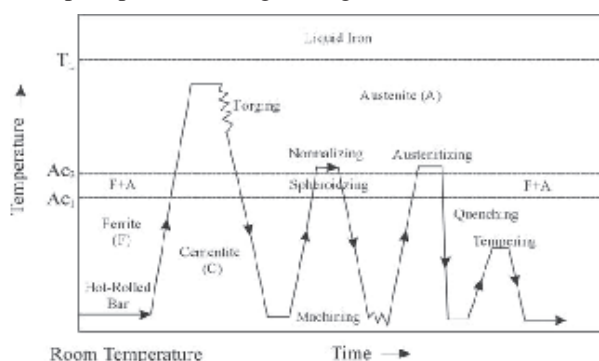


Fig. 1 : Temperature-time processing schedules for producing quench and tempered forgings<sup>[2]</sup>.

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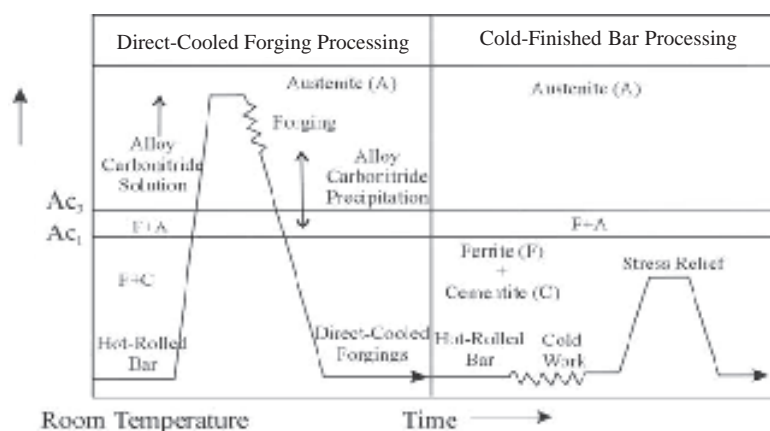


Fig. 2 : Temperature - time schedules for producing direct cooled microalloyed forgings and cold finished bars<sup>[2]</sup>.

cooling from as forged shape change. Therefore vanadium bearing steels has eliminated number of processing steps thereby reducing cost of heat treatment, material handling and equipment costs. Following Fig. 1 has showed processing steps associated with quench and tempered state normal forging steel and Fig. 2 has showed processing step of microalloyed forging steels<sup>[2]</sup>.

The mechanism of vanadium carbonitride precipitation has been inter-phase precipitation. This has been interfaces between austenite and ferrite during phase transformation when cooling as forged products through phase transition lines. The interface of austenite and ferrite leaves array of embedded precipitates in growing ferrite phase at a decrease in size of austenite grains. Fig. 3 has showed vanadium carbonitride precipitation of 10 nm size in an as forged complex shaped steel product made of 0.2% C - 0.14% V steel composition.

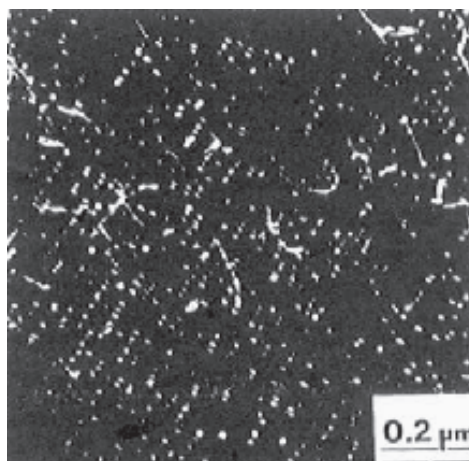


Fig. 3 : Fine vanadium carbonitride precipitates in ferrite of a 0.2 wt.% C - 0.14 wt.% V steel air cooled from 1473 K. Figure has showed a dark field transmission electron micrograph<sup>[2]</sup>

Precipitation reaction has taken place between a substitutional impurity (V, Nb) and an interstitial impurity (C, N) of austenite solid solution. During heating VN has dissolved rapidly than NbN. Figure 4 has showed austenite grain coarsening behaviour of steels containing V, Al, Nb,

and Ti and has showed that grain coarsening in Ti-containing steels does not occur at temperatures around 1473 K or higher.

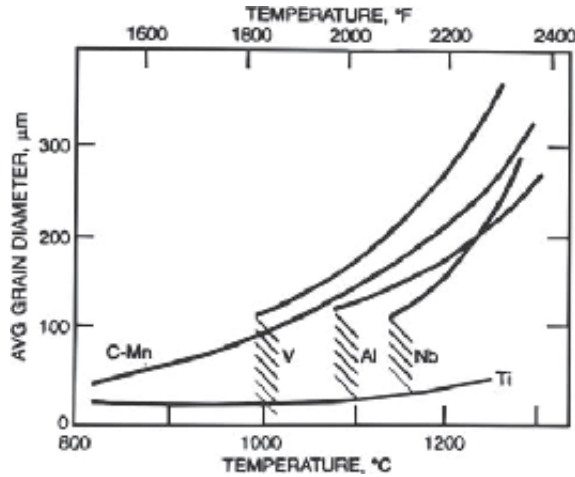


Fig. 4 : The effect of various microalloying elements on austenite coarsening<sup>[2]</sup>.

**Hot working conditions in vanadium microalloyed steel<sup>[3]</sup>**

Evidences of improvement in properties by vanadium microalloying have been as follows. (a) Increases in yield strength by 8% have been observed for every 0.1% V increment, (b) Increasing vanadium from 0.062% to 0.11% the mean flow stress has increased from 12 to 25%, (c) 0.09% vanadium microalloyed steel has showed 30% increase in flow stress over that of aluminium killed steel. Investigations have been done about effects of vanadium on flow stress in microalloyed steel during hot rolling. Modeling of hot rolling has been studied to know the strain for dynamic recrystallization. Simulations of hot strip mill have been done by Phaniraj and Lahiri by compression tests at similar strain and temperature. Neural net work model has been developed to compare with semi-empirical methods. Modeling has predicted the dynamic recrystallization. Chemical composition of the alloy has been shown in Table 2.

Table 2 : Chemical composition of vanadium microalloyed steel<sup>[2]</sup>.

C	Mn	Si	Al	N	V	S	P	Fe
0.073	0.74	0.084	0.048	0.0059	0.065	0.007	0.01	Balance

**Warm forging of vanadium and titanium microalloyed steel<sup>[4]</sup>**

Titanium and vanadium microalloyed medium carbon steel has been studied for mechanical industry. The properties of interest have been (a) continuous cooling diagram, (b) hot and warm ductility, (c) grain growth, (d) static and dynamic recrystallization, and (e) effects on the microstructures. In order to improve toughness properties titanium has been added along with vanadium. Titanium - vanadium has precipitation hardening capacity to control degree of grain growth and recrystallized austenite grain size. The context have investigated minimum titanium requirement for microalloying to refine ferrite and pearlite structure, without affecting other properties. Warm forging has been forging at  $\alpha + \gamma$  regions of phase diagram. Aim of warm forging has been (a) to remove heat treatment step (b) to achieve optimum mechanical properties,



(c) good dimensional precision similar to cold forged parts (d) good surface finish containing thin oxide scale (e) elimination of machining process to produce finished products and (f) decreased requirement of fuel, furnaces and electrical power for fabrication. This has been accounted as economy in fabrication by about 50%. Warm forging has permitted hardness increment with decrease in forging temperature. Grain sizes have dropped with temperature decrease. The smallest grain sizes has been achieved at about 0.019% titanium. Rise in deformation temperature has increased the pearlite volume fraction. Fineness of grains and comparable temperature variation in warm forging has produced balanced properties in microalloyed steel, e.g. complementing behaviour between strength and toughness. Raising the temperature of forging above 1323 K have increased strength of components, however surface finish deteriorates. Vanadium and titanium microalloyed steel under warm forging between 1023 and 1048 K has showed increase in ductility. Ductility has appeared to increase further with rate of deformation at these temperatures. As for example constant velocity universal joint-an automotive component has warm forged close to 1043 K to obtain best mechanical properties with enough warm ductility. Chemical composition has been shown in Table 3.

Table 3 : Chemical composition in weight percentage and parts per million (p.p.m.)<sup>[3]</sup>.

Steel No.	%C	%Mn	%Si	%S	%P	%Cr	%Ni	%Mo	%V	%Ti	%Cu	%Sn	%Al	N2, p.p.m.	O2, p.p.m.
1	0.29	1.34	0.41	0.026	0.021	0.09	0.10	0.02	0.1	0.003	0.244	0.02	0.029	167	30
2	0.29	1.28	0.34	0.028	0.017	0.13	0.08	0.01	0.09	0.019	0.134	0.015	0.036	106	45
3	0.32	1.39	0.33	0.021	0.015	0.13	0.14	0.03	0.129	0.039	0.129	0.017	0.049	148	57

#### Thermo-mechanical treatment of niobium and vanadium microalloyed steel<sup>[5]</sup>

Fine and uniform microstructure has been produced by thermomechanical treatment by utilizing kinetics of recrystallization of hot working process. Improvements in features required by thermal-mechanical processing have been listed as follows: (i) control of initial austenite grain size, (ii) volume of carbides and nitrides in solution, (iii) required mechanical treatment, (iv) balances in composition and (v) inter-pass time in processing. Family of microalloyed steels has contained either single or combination of niobium, vanadium, molybdenum, titanium, boron or aluminium. Advantages of micro-additions have been (i) strengthening through precipitation, (ii) grain refinement. (iii) retardation of recovery and recrystallization and (iv) control of transformation kinetics, e.g. bainitic transformation for achieving yield strength upto 700MPa for automotive components. Austenite grains at all temperatures upto 1473 K have been refined by increasing niobium content between 0.06 to 1.03%. Coarsening of grains have appeared beyond stoichiometric composition of Nb:C. The restoration process has been retarded by solute drag at higher temperature while strain induced precipitation of carbonitrides at lower temperature. During reheating process carbides has remained in solution in austenite which on cooling has formed fine, planer coherent precipitation in ferrite matrix. Roll reduction at different strain rates have been described as follows: (i) Precipitation have caused increased retardation of dynamic recrystallization at lower strain rates when Nb content has been 0.073%. Further retardation of beginning of dynamic recrystallization has been caused by addition of 0.14% vanadium at a strain rate of 13 sec<sup>-1</sup>. Optimization of multistage plane strain compression in plate rolling schedules has referred to form complete recrystallization to pan caking of microalloyed steels containing 0.118 wt.% Nb. Niobium has been found to increase compressive

load above three roll reduction passes accounted to be 70%. Modification of softening behaviour has been stated to be due to solute effect, strain induced precipitation and combination of both. Precipitations have been found to retard the dynamic recrystallization. Dynamic precipitation has been studied under coexistence of Mn, Nb and V. Mn content increase has reduced carbonitride precipitation. Therefore kinetics of precipitation has been retarded by other elements. At about 1073 K high niobium and high vanadium has been found to reduce the ductility by formation of edge cracking. Different responses of high Nb and high V have been studied at high temperature, constant true strain rate, single and multi-stage compression. These responses have been (i) unusual display of flow curve in comparison to conventional alloy behaviour, (ii) retard grain growth, (iii) retard dynamic recrystallization, (iv) high rates of precipitation and (v) increase in related hardening effects. Chemical composition has been shown in Table 4.

Table 4 : Chemical composition of material (wt.%)<sup>[5]</sup>.

C	Mn	Si	Nb	V	Cu	Ni	P	Ti	Ca	S	N
0.1	1.093	0.3645	0.0877	0.0795	0.0152	0.0138	0.0076	0.0042	0.0032	0.0023	0.0092

#### Heat treatment of vanadium modified alloy steel<sup>[6]</sup>

Strategic developments of microalloyed steels and related heat-treatments have improved properties of microalloyed steels. Selections of suitable combination of alloy elements have been responsible for improved properties of steel. Increasing demands of steels have produced standard grades of steel for applications in low stressed and highly stressed zones of application. As for example, highly stressed components have been power transmission gears and shafting, ball and roller bearing, and spring members.

Better hardenabilities have been achieved by modifications of alloy steels with 0.10 - 0.15% V additions than standard Mo-containing alloy steel grades. When vanadium is used singly the austenitizing temperature increases. However, austenitizing temperature has not been increased in medium carbon steels when both elements are used together. Better hardenability has appeared by favourable interaction between vanadium and molybdenum. These have exceeded hardenability of 0.3% Mo-steel. Table 5 has showed chemical composition of steel.

Table 5 : Composition of alloy steel (wt.%)<sup>[6]</sup>.

Alloy	C	Mn	P	S	Si	Ni	Cr	Mo	V	Al	N
STD 4330	0.3	0.49	0.003	0.017	0.21	1.78	0.53	0.30	< 0.01	0.056	0.0135
Partial V sub stitu tion, 4330	0.28	0.51	0.005	0.009	0.23	1.93	0.53	0.05	0.15	0.065	0.0069
	0.28	0.49	0.004	0.016	0.20	1.94	0.53	0.10	0.10	0.067	0.0081
	0.30	0.48	0.001	0.014	0.22	1.76	0.53	0.10	0.16	0.063	0.0117
Total V substi tution, 4330	0.31	0.50	0.001	0.016	0.23	1.72	0.50	< 0.01	0.24	0.056	0.0134
	0.29	0.52	0.003	0.017	0.21	1.75	0.53	< 0.01	0.15	0.053	0.0082
	0.28	0.50	0.004	0.011	0.21	1.94	0.52	< 0.01	0.10	0.058	0.0048



**Influence of vanadium on mechanical properties of air hardening steel<sup>[7]</sup>**

Air hardening steels have wide application because of wear resistance and high toughness linked to high carbon content. However these steels have low impact toughness. To improve impact toughness micro-additions of vanadium has been done within range of 0.5 - 3.0%. Cast microstructure has showed primary austenite dendrite morphology. Increase in vanadium content has lowered hardness and improved impact toughness to produce appropriate combination of toughness and hardness, or abrasive and impact-fatigue-wearing resistance. Decrease in hardness and improvement in impact toughness has been produced by martensite and retained austenite fine structure after heat treatment. Increase of vanadium content from 0.523 to 2.990%, the hardness has decreased from 61.5 to 57.2 HRC and associated increase in impact toughness has been from 3.48 to 8.35% (Fig. 5). Presence of hard carbides type  $(Cr, Fe)_7C_3$ ,  $V_6C_5$  and VC, their content and morphology obtained has enabled good wear resistance property.

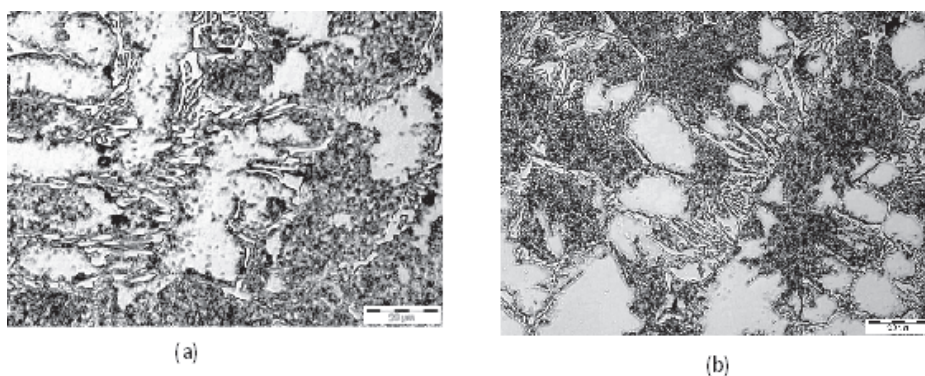


Fig. 5 : Microstructure of alloy at (a) low vanadium content and (b) high vanadium content in air hardened-microalloyed steel <sup>[7]</sup>.

fluence of vanadium on structure, hardness and impact toughness has been investigated on X180CrMo12-1 alloys. Activity of vanadium in alloy has been (i) to move liquidous and solidous lines to the higher temperature, (ii) to form  $V_6C_5$  carbides with proper distribution within  $(Cr, Fe)_7C_3$  carbides and austenite, (iii) growth of austenite dendrites followed by precipitation (iv) to form fine grained metallic base, and (v) to enable formation of  $(Cr, Fe)_23C_6$  carbide and its precipitation in austenite during cooling process, thereby ease in formation of martensite and (vi) to reduce retained austenite. Under satisfactory hardness level noticeable improvement in impact toughness has been observed by even trace of vanadium. Table 6 has showed impact toughness, hardness and chemical composition of steel.

Table 6 : Hardness and impact toughness of specimens<sup>[7]</sup>.

Sl. No.	Impact toughness, [J/cm <sup>2</sup> ]	Hardness (HRC)	Chemical composition				
			C [%]	Cr [%]	Mo [%]	S [%]	V [%]
1	3.48	61.5	1.801	11.754	1.298	0.021	0.523
2	3.59	60	1.766	11.556	1.301	0.023	1.005
3	3.71	59	1.732	11.466	1.268	0.022	1.998
4	8.35	57.3	1.762	12.236	1.248	0.02	2.990

**Effect of heat treatment on microstructure and tensile properties of ultrafine grained steel<sup>[8]</sup>**

Ultrahigh strength and enhanced toughness of ultra fine grained (UFG) C-Mn steel have great potential as advanced material. These alloys have been inferior with reference to strain hardening capability and ductility than coarse grained alloys when exposed to severe plastic deformation. Rapid dynamic recovery during deformation of ultrafine grained steel has lowered strain hardening capability. Employment of effective obstacles to dislocation motion within grains has improved strain hardening effects. Improvement of thermal stability and strain hardening capability has appeared by embedding vanadium precipitates in matrix of UFG ferrite. Severe deformation occurs by equal channel angular pressing (ECAP). Effective precipitate presence has pinned dislocation to improve strain hardening effects, however at lower V content this effect has not been appeared. This context has described effects on strain hardening by severe plastic deformation and design of heat treatment schedule. Steels containing 0.34% vanadium has been treated (i) by conventional normalization for precipitation before ECAP and (ii) by isothermal transformation for vanadium precipitation during ECAP and subsequent annealing. Normalized precipitates before ECAP has appeared to coarsen when annealed after ECAP, whereas precipitation during ECAP followed by annealing has showed stability in precipitate morphologies. Improvement in strength and strain hardening capability has arisen from severe plastic strain induced nano-sized precipitation in ultrafine ferrite grains. These have interacted with the dislocations for strengthening. The tensile properties have been shown in following Table 7.

Table 7 : Tensile properties of as (i) normalized+ ECAP + anneal and (ii) ECAP + anneal specimen<sup>[8]</sup>.

Conditions		YS, MPa	UTS, MPa	Uniform elongation	Elongation to failure
Normalized+ ECAP + anneal	Before ECAP	435	568	17	28
	After ECAP	920	920	2	9
	anneal at 873 K, 1 hr.	441	516	18	31
ECAP + anneal	Before ECAP	465	643	14	18
	After ECAP	925	949	2	10
	anneal at 933 K, 1 hr.	718	796	9	19

**Tempering processes /technology<sup>[9]</sup>**

Conventional tempering processes have been heating hardened steel below lower critical temperature at a suitable rate to increase (i) ductility, (ii) toughness, (iii) grain size, (iv) to relieve quenching stresses, (v) to ensure dimensional stability, (vi) reduce hardness of welded zones and (vii) relieve stresses of forming and machining. Variable associated to affect microstructure and mechanical properties of steel by tempering treatment have been (i) tempering temperature, (ii) time of tempering, (iii) cooling rate from the tempering temperature and (iv) composition of steel, i.e. carbon content, alloy content and residual elements. Distinct changes in microstructure by tempering have been stage I: (373-523 K) formation of transition carbide and lowering of carbon in martensite upto 0.25%, Stage II: (473-573 K) transformation

of retained austenite to ferrite and cementite, Stage III: (523-623 K) replacement of transition carbide and low temperature martensite by ferrite and cementite. Increase in hardenability has been main purpose for addition of alloying elements.

Alloying elements in steel has reduced softening rate on tempering at higher temperature. Alloying elements have been two types (a) Non-carbide formers and (b) Carbide formers. Nickel, silicon, aluminium, and manganese have been non-carbide formers and remain in ferrite as solid solution, whereas chromium, molybdenum, tungsten, vanadium, tantalum, niobium, and titanium has been carbide formers, these have retarded softening tendency during tempering. Chromium, vanadium and molybdenum have been strong carbide. Former has increased hardness above 478 K. Phosphorous, nickel and silicon has attributed solid solution strengthening. Carbide formers have retarded cementite grain coalescence during tempering. High alloy steels have showed secondary hardening effects under certain conditions.

#### Tempering behaviour of steel<sup>[10]</sup>

0.1% C-9%Cr-1%Mo-0.2%V steel has tempered at 1023 K for 100 hours. Intermediary observations have been as follows. Activation energy for recovery of martensite has been sum of activation energy for self-diffusion of iron and the activation energy of boundary diffusion of substitutional elements. Activation energy of self diffusion has been necessary for climbing and activation energy for boundary diffusion has been necessary for decomposition of M<sub>23</sub>C<sub>6</sub> precipitated on the boundary. In normalized state ultrafine grains of 0.1  $\mu$ m size has surrounded the boundaries of martensite lath and blocks. Through coalescence of martensite and ultrafine grains, subgrains have formed with decrease in the number of ultrafine grains. Ten hours of tempering have showed discontinuous changes in preferred orientation, lattice strain, number of ultrafine grains and average crystalline grain sizes. Dissolution of M<sub>23</sub>C<sub>6</sub> precipitates at dislocations during tempering has taken place by annihilation of dislocations being induced by martensite transformation and formation of subgrains. Particle size has been reduced by second process of dissolution. A depression has appeared at the middle of isothermal tempering curve. Combination of changes in lattice strain, number of ultrafine grains and precipitation of M<sub>23</sub>C<sub>6</sub> has been explained by the depression. Growth rate of M<sub>23</sub>C<sub>6</sub> has been lowered by time exponent of 1/16 due to dissolution of carbides during advance of tempering. Development of high chromium heat resistant steel has improved life of components required for power generation; however creep resistance has dropped after 10,000 hours of exposure. This depends on chemical composition and heat treatment. Formation of z-phase accompanying the dissolution of MX type precipitates and local recovery around prior austenite grain sizes have been the reasons for such behaviour. Abnormal phenomena observed in modified chromium steel, like increase in hardness after 20,000 hours of creep exposure at 923 K and drop in creep strength has been studied through tempering behaviour at greater temperatures. Different ways of study on similar incidences have focused role of strong carbo-nitride formers, i.e. V, Nb, Ta and Ti, because these elements have formed stable fine precipitates of an MX type, that has resulted in retarding recovery. Interaction between MX precipitates and dislocations have precipitated at early stage of tempering than redissolution on further tempering. These further have been precipitated by subsequent tempering to retard growth of precipitates and thus have increased creep life. Dissolution of precipitates has occurred by M<sub>23</sub>C<sub>6</sub> precipitates along with MX precipitates. Table 8 has showed chemical composition of steel.

Table 8 : Chemical composition of experimental steel (mass %)<sup>[10]</sup>.

C	Si	Mn	P	S	Cr	Mo	V	N	Al	Fe
0.1	0.3	0.39	0.011	0.0006	9.04	0.99	0.189	0.033	0.017	Bal.

**Dissolution of vanadium nitride during tempering<sup>[11]</sup>**

Both high temperature strength and toughness have improved by addition of small carbonitride formers to full martensitic heat resistant steel. Studies of precipitation behaviour in martensitic steel and interaction with dislocations have described longer duration for rupture time microalloyed steel in contrast to plain carbon manganese steel. These have been complex interplay of carbonitride formers. These have been complex MX type precipitates, M<sub>23</sub>C<sub>6</sub> and Laves phases. Studies on precipitation behaviour of VN in high chromium steel have referred to following observations. Re-precipitation and redissolution consequences of VN precipitates during isothermal tempering at 1013-1073 K on 7%Cr-0.4%V-0.09%N steel have showed that VN precipitates at early stage of tempering and then a quasi-steady period when VN precipitates redissolve partially and further reprecipitate again. Redissolution of VN has been confirmed by temporal decrease in hardness and temporal increase in integral breadth of a {110} X-ray diffraction peak. Fine reprecipitation has appeared at ferrite subgrains and on sub-grain boundaries after appearance of diffraction peak. 0.14%C-9%Cr-1%Mo-0.2%V-0.09%Nb alloy has showed similar effects in case of niobium carbides. As stated previously the mechanism of redissolution of precipitates have been (i) Annihilation of dislocations and reprecipitation of MX type particles on dislocation progressing parallel, (ii) Formation of quasi equilibrium state by pinning dislocation at saturation, (iii) Pinned dislocation have been unlocked by mechanism of local climb of dislocations, which in absence of thermodynamic equilibrium redissolve and tempered martensite has transformed to ferrites. MX precipitates has further formed in the ferrite grains and subgrain boundaries. New ferrite grains have produced new local stresses around grains to induce unlocking of pinned dislocations. These chain reactions have produced sharp peak for MX redissolution.

**Micro-magnetic NDE of isochronally tempered steel<sup>[12]</sup>**

Developments have appeared about new methods for inspection and life assessment of light water reactor plant. Aging of components have caused materials degradation over time to reduce life of components. Degree of aging have termed as damaged state of material, which has subjected to remaining life assessment after verification of internal conditions. Reactor pressure vessel steels those have affected by radiation embrittlement have been inspected by non-destructive evaluation (NDE) techniques. However NDE techniques have been deficient in detection of changes in precipitate phases in stainless steel. Mechanical properties have been influenced by microstructural imperfections, e.g. dislocations, segregates, inclusions, grain boundaries and stresses. These crystal imperfections have varied some magnetic properties like hysteresis, coercive force and remanence. Under an axial magnetic force, defects have prohibited the movement of Bloch walls and reorientations of magnetic domains. Defective material magnetization has produced discontinuous changes in magnetization, generate Barkhausen noise (BN) and time derivative of actual magnetic flux into a coil placed near the object. Martensitic stainless steel containing 12% chromium has been used upto 800K because of their non-oxidizing and superior high temperature mechanical properties for electrical power generation. This context has simulated ageing behavior by heat treatment experiments. Effects

of austenitizing temperature on the structure-sensitive magnetic properties and BN characteristics has investigated as a function of isochronal tempering temperature. At a tempering temperature of 773K, for one hour followed by over-aging has showed secondary hardening effects with hardness increment to 47.5 HRC. A slight increase in the structure sensitive magnetic properties (Hc, Br and Wh) has been observed as first effect of tempering regardless of austenitizing temperature. This has followed by rapid magnetic softening, with a minimum at 1073 K. From 1073 K, by passing Curie temperature of 1041 K for pure iron abrupt increase has been found in magnetic properties. An inverse relation to that of structure-sensitive magnetic properties has been exhibited by variation in BN energy with tempering temperature. Coarsening of precipitates by Ostwald ripening, regardless of austenitizing temperature has been cause of rapid increase of BN energy upto 1023 K. The mechanical properties, like hardness associated with microstructural evolution has suggested this, micromagnetic technique as a non-destructive evaluation technique provided linear relation between BN energy and hardness has existed. Chemical composition of steel has been shown in table 9.

Table 9 : Chemical composition of as-received 12% CrMoV steel<sup>[12]</sup>.

C	Cr	Mo	Mn	Ni	Si	V	Nb	P	S	Fe
0.18	10.81	0.90	0.68	0.55	0.22	0.19	0.051	0.009	0.002	Balance

#### DTBT of ferritic-martensitic steel<sup>[13]</sup>

Potential candidate for blanket and first wall structure of fusion reactor has been ferritic-martensitic steels containing 9 - 11 wt.% Cr because of their high strength, low thermal dilatation and high resistance to void swelling. Sensitivity to embrittlement under neutron or proton radiations of fusion reaction has been characterized by ductile to brittle transition temperature (DBTT) to higher values. Evaluation of DBTT has been done by instrumented Charpy tests at varying temperatures.

Evaluation strategies have referred to a dynamic quasi-equilibrium approach (DQEA). DQEA has ensured that the impact force of pendulum, acting on the specimen parallel to the notch tip, has approximated to a one-dimensional line force that has increased steadily with time before brittle failure. DQEA has enabled evaluation of dynamic Weibull moduli ( $m$ ) and consequently, dynamic Weibull master curves expressed in terms of (i) initial defect sizes, if no stable (micro)crack growth starting from initial defects has occurred before brittle failure, or (ii) critical crack size, if stable (micro)crack propagation takes place before brittle fracture. Dynamic capacity of a solid to undergo stable (micro)cracking and crack tip shielding before brittle failure has estimated from dynamic Weibull master curves. M.Lambrigger has experienced dynamic Weibull master curve for specimens of normal size and subsize (10CrMoVNb Manet II alloy). These Weibull master curves have been used to evaluate factors for prediction of DBTT shifts of ferritic steels resulting from irradiation embrittlement. The factors have been capacity of shielding and stable (micro) crack before brittle failure. If steel has showed high capacity to undergo stable (micro) cracking before brittle failure as well as good capacity for shielding crack tips of large and medium size cracks in the absence of plasticity, a reduction has found in DBTT shifts due to irradiation embrittlement. Toughening mechanism has appeared to control stable propagation of mean size (micro) crack, which has arisen from only normal sized specimens of Manet II. However, the case has subject to traditional concept of capacity to undergo stable microcracking and crack tip shielding in relation to capacity to shield in



presence of full matrix plasticity, quantified by absorbed impact energies of Charpy tests carried out in the high temperature range. The absence of matrix plasticity has been characteristic to lower DBTT range. Irradiation induced DBTT shifts has linked to subsized specimens on Manet II, where crack tip shielding of large and medium size cracks at low homologous temperature has been observed that has been quantified by toughening exponent in dynamic tests. Dynamic shielding has appeared for large critical crack sizes.

#### **Evaluation of irradiation effects<sup>[14]</sup>**

To develop materials (12Cr1MoV steel) for fusion reactor under irradiation effects small-scale specimens have been tested using an ion irradiation facility. Estimation of ductile to brittle transition temperature (DBTT) has been done by small punch (SP) test in smaller specimens made from broken Charpy impact test. A unique relation in DBTT has been found between test results of Charpy V-notch (CVN) and SP test results. Limitations of SP test have arisen for strongly anisotropic materials and information about direction of crack growth. Therefore SP test has appeared to be limited within narrow range of materials such that perturbations appear to be similar between both types of test results. Evaluation of effects by irradiation have been described after many experiments. As for example 16 MeV proton irradiation effect has simulated to 14 MeV neutron damage energy spectrum over most of the energy range.

#### **Tempered martensitic embrittlement in steel<sup>[15]</sup>**

To achieve higher plasticity and toughness as well as to keep sufficient hardness low temperature tempering of steels has been done with previous martensite microstructure within temperature range of 483 - 673 K. Above tempering temperature of 473 K a decrease in toughness has been observed. This phenomenon is defined as tempered martensite embrittlement (TME) or low tempering temperature brittleness or martensite embrittlement. TME has been described to associate intergranular fracture; however other kinds of fracture such as cleavage, quasicleavage, fibrous fracture or mixed fracture have also appeared. Broadly fracture in TME has been classified as intergranular and intragranular. The formation of interlath carbides from decomposition of retained austenite has attributed transgranular mode of fracture. This mode has exerted from surface active impurities and grain boundary carbides. Evidences have referred to absorption of impurities by carbides initially and then have released from carbide crystal lattice to the interfaces of carbide and matrix to segregate impurities for initiation of this crack. Evidence has described impurity segregation from austenitization state; which on enrichment upto certain level has initiated transgranular cracks. In any case this has been to lower critical stress requirement for brittle crack formation and its growth. No evidence has been found to describe strain aspect of embrittlement.

Tempered martensite embrittlement (TME) has been investigated in low alloy 3Cr-Mo-V steel within temperature range of 523 - 723 K. The observations have been (i) appearance of TME has been 623 K, (ii) TME fracture mechanism has been transgranular cleavage in place of intergranular fracture, (iii) retained austenite in as quenched state has been observed to decompose during tempering to introduce cleavage fracture in a transgranular mode, (iv) presence of fine particles inside the grains have been another cause of transgranular fracture during TME. Chemical composition of steel has been shown in table 10.



Table 10 : Chemical composition of steel, (wt. %)<sup>[15]</sup>.

C	Si	Mn	Cr	Mo	V	Ni	Cu	Al	S	P	Sn	Sb	As	Fe
0.14	0.21	0.5	2.93	0.6	0.29	0.14	0.11	0.014	0.019	0.013	0.12	0.002	0.002	Bal.

**Z-phase formation in martensitic steels<sup>[16]</sup>**

A series of 12CrMoVNb martensitic steels has been investigated for long term creep rupture studies to use in high temperature components in steam and gas turbines above 823 K. Microstructural instability of these alloys have been manifested by rapid reduction in creep rupture strength and corresponding increase in creep rupture ductility. The microstructural instability by creep exposure has been precipitation, coarsening and dissolution of various carbides and other phases. Studies of creep behaviour in 12CrMoVNb alloy steel within 823-873 K and time limit of 1,00,000 hours have been described as follows: Minor phase evolution in high temperature creep resistant martensite 12CrMoVNb alloy steel after heat-treatment has been formation of primary phases like NbX, M<sub>23</sub>C<sub>6</sub>, Laves phases (Fe<sub>2</sub>Mo) and compositions of complex nitrides, Z-phase and Cr(V, Nb)N. Modified Z-phase has been a complex nitride phase rich in chromium, vanadium and niobium. The unit cell has been tetragonal. Dissolving primary NbX particles have associated modified Z-phase. Less stable M<sub>2</sub>X and MX nitrogen rich phases have been replaced by modified Z-phases as an equilibrium nitride phase within temperatures of 823-873 K in 12CrMoVNb alloy steel. Z-phase has reduced creep rupture strength of this steel by removal of precipitation strengthening elements such as Cr, V, Nb and N from the matrix. Table 11 has showed the creep rupture data.

Table 11 : Creep ruptures data on 12CrMoVNb steel<sup>[16]</sup>.

Test piece	Temperature, °C	Stress, MNm <sup>-2</sup>	Creep rupture life, h	Elongation, %
A	600	309	300	3.6
B	600	201	9200	2.5
C	600	139	22,050	22.5
D	600	100	30,800	30.0
E	600	69	94,800	24.0
F	550	201	79,200	14.0

**Compositional changes by thermal exposure<sup>[17]</sup>**

Assessment of time-temperature history of creep resistant power plant steels has been considered promising when studies have made about compositional changes in the minor phases during service exposure. Tempering in heat-treatment processes has changed chemical composition of equilibrium minor phases. Variation of service temperature and heat-treatment temperature has therefore varied chemical composition in proportion. Therefore time-temperature history after prolonged service exposure has been achieved from chemical composition variation of minor phases. In all classes of (9-12)% Cr steels M<sub>23</sub>C<sub>6</sub> and M<sub>2</sub>X phase has been accepted as most suitable time-temperature indicator as it has been thermodynamically more stable. On the other hand no change has been found in MX precipitates, where service temperature (823-873 K) has been lower than tempering temperature. In addition, equilibrium precipitation of carbides and nitrides during service exposure has proved to be a suitable marker of thermal history of components.

**Stress relief crackling in advanced steel<sup>[18]</sup>**

Desulphurisation of hydrocarbon or cracking of heavier hydrocarbon to lighter molecules have been carried out in reactors and pressure vessels of refinery. High temperature (723 K), pressure (100 kg/cm<sup>2</sup>) and hydrogen environment are required for these processing in presence of catalyst. Various steel generations have developed to serve these purposes. Vanadium modified Cr, Mo alloy have developed as 5th generation of steel in 1995. Forgings or plates have been manufactured to produce equipments of large wall thickness. Cracking during services have appeared due to reheat, or relaxation, or shut down, or repair in welds. Service embrittlement of heat resistant steels, e.g. reheat cracking has been as follows: (i) faster heating between 773-973 K during stress relieve annealing, (ii) welding parameters including interpass temperature control while welding, (iii) Ratio of chromium and vanadium which form carbide susceptible to reheat cracking, (iv) Welding diameter. Lower diameter has provided lesser heat input during inter-pass. Crack in weld metal has oriented along both longitudinal and transverse direction to that of direction of welding. Crack in heat affected zone (HAZ) has been always parallel to direction of welding (Fig. 6).

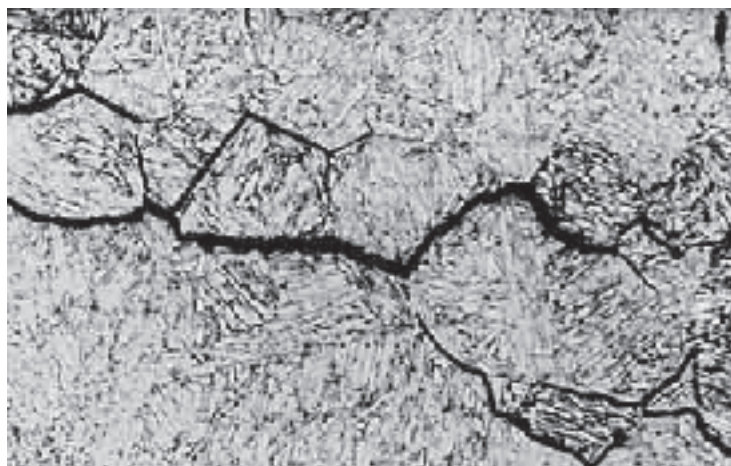


Fig. 6 : Marocrack with branches along prior austenite grain boundaries<sup>[18]</sup>.

**Reheating cracking in steel<sup>[19]</sup>**

Post weld stress relief heat-treatment within 623-823 K in microalloyed steels containing chromium, molybdenum and vanadium has produced reheat cracking. Cracking has appeared in coarse grained regions of heat affected zone and weldment. Cracking has been in the form of macro-cracks or colonies of micro-cracks.

Principal cause for cracking has been relaxation of residual stresses to grain boundaries for creep deformation, where grain interior has been strengthened by carbide precipitation. Prevention of reheat crack formation has been (i) choice of steel that has not been susceptible to reheat cracking, (ii) Choice of steel having low level of impurity elements, e.g. Sb, As, Sn, S and P. (iii) Refinement of HAZ has been promoted by buttering a thin weld metal layer using thin electrodes (3.2 mm) followed by larger diameter (4-4.8 mm) electrode so as to generate more heat to refine any coarse grain, (iv) avoid stress concentration, e.g. grind weld toes with preheat maintained.

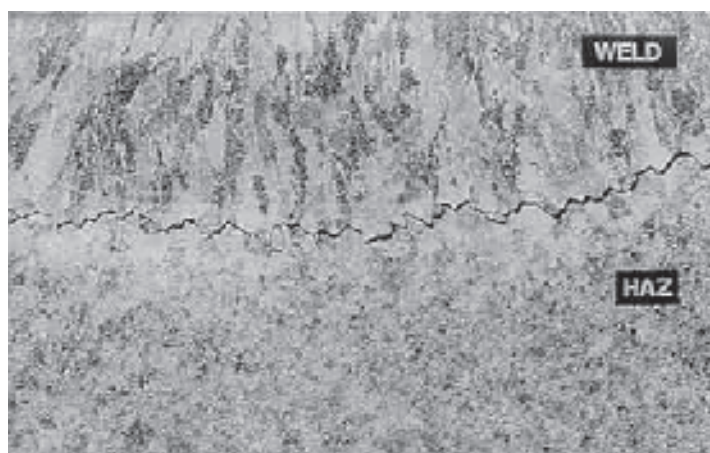


Fig. 7 : Cracking by post weld heat-treatment<sup>[19]</sup>.

#### Properties of martensitic stainless steel<sup>[20]</sup>

Knife and cutlery steel have been made of martensitic stainless steel containing 12-18% chromium. Significant influence has observed regarding matrix composition of Cr and C in stainless steel when exposed to heat treatment schedules. A new type martensitic stainless steel 6Cr15MoV has been developed after consideration of better hardness and corrosion resistance. The equilibrium carbide in these steel have been  $M_{23}C_6$  carbides, these carbides have found widely distributed in the matrix under annealed conditions. This martensitic stainless steel has showed about 60.8 to 61.6 HRC hardness when quenched from 1323-1373 K. Carbides have resisted austenite grain growth at high temperature. Tempering after quenching at 373-423 K have retained hardness about 59.2 - 61.6 HRC and Charpy U-notch impact toughness about 17.3-20 J. This treatment has been accepted to produce excellent comprehensive mechanical properties in this stainless steel. Increasing tempering temperature to 773-813 K has precipitated  $M_{23}C_6$  carbides at the martensite lath boundaries and reduction in impact toughness.  $M_3C$  precipitates have existed in martensite matrix of test steel after tempering at 773 K. These precipitate sizes have been bigger than  $M_{23}C_6$  precipitates.

#### Performance of ferritic stainless steel<sup>[21]</sup>

Popular grades of traditional stainless steels (e.g. 304, 316) has been alloyed with nickel (8-14%) to increase corrosion resistance in reducing atmosphere and to serve as austenite former and subsequently to balance ferrite forming elements, e.g. chromium. Although popular grade but these stainless steels suffer from pitting corrosion, crevice corrosion, halide induced stress corrosion cracking (SCC) and incidences of corrosion fatigue in relatively mild halide environment. Modern stainless steels to resist localized corrosion have been high alloyed duplex - 2205, austenitic - 254SMO and superduplex - 2507. Ranks of these steels have been formulated by pitting resistance equivalents (PREs). Absence of molybdenum has reduced brittleness induced from intermetallics. Therefore removal of molybdenum has reduced stress corrosion cracking. Edge cracking effects or low hot ductility has appeared in duplex stainless steel alloys than austenitic grades during hot rolling operations. Vanadium has been considered as carbide former to improve high temperature properties by precipitation strengthening. A major beneficial effect of vanadium on 18% Cr ferritic stainless steel has been described with regard

to corrosion, mechanical and formability properties. Increase in corrosion resistance by vanadium addition has competed to equivalent performance of nickel. As heat exchanger tubes 18Cr-4V alloy has showed potential application, which has been prone to pitting, SCC and corrosion fatigue.

#### **Metallographic examination of HAZ<sup>[22]</sup>**

High pressure and high temperature components in power plants and chemical industry have been great consumer of X20CrMoV 12 1 steel. Importance of this case has been similar mechanical properties, chemistry and microstructure of the base metal and weldment. Post weld heat-treatment has been accepted as important processing step for developing these effects. Heat affected zone (HAZ) in weld adjacent to the fusion boundary has showed martensite phases by metallographic tests and micro-hardness tests. In addition to standard post weld heat-treatment, an additional tempering heat treatment has been applied and observed to transform these martensite phases to ferrite and carbide. Therefore equalization of properties has been possible.

#### **Effect of V and Nb on HAZ of steel<sup>[23]</sup>**

Thermal cycles experienced during welding have produced poor toughness in the heat affected zone (HAZ). This has upset balance of high strength and good toughness in HSLA steels. Adjacent to the weld fusion line is called heat affected zone (HAZ), which has been a part of weld. In HAZ grain coarsened, HAZ has been zone of lowest toughness. This zone has picked up peak temperature equal to melting point to coarsen austenite grains. This has followed by rapid cooling to promote brittle microstructure made of ferrite side-plates. Further investigation have referred to that inter critically grain coarsened heat affected zone (IC GC HAZ) has been the most degraded part of HAZ. This has formed from reheating GC HAZ regions between critical temperatures Ac1 and Ac3 during subsequent welding passes. Segregation of carbon or manganese has formed austenite blisters within inter-critical temperatures. These zones has formed martensite-austenite (M-A) phases on cooling to reduce HAZ toughness. Microstructural simulation of coarse grained heat affected zone (GC HAZ) has been done on boron free four types of steel. These are C-Mn-0.5V, C-Mn-0.11V, C-Mn and C-Mn-0.03Nb steels. The processing has involved reheating to 1623 K, rapid cooling to room temperature and further reheating to either 1023-1073 K. Toughness of specimens have been assessed by Charpy tests. Simulated specimens have been tested in scanning electron microscope (SEM) to assess martensite-austenite phase field, their distribution and area fraction. Increasing micro additions has increased M-A phase fields to decrease toughness. Addition of 0.05% V to C-Mn steel has resulted in the lowest Charpy transition temperature. Toughness has appeared to deteriorate with rising level of vanadium to 0.11%. This has accounted from (i) increased martensite-austenite (MA) phase (ii) larger average and maximum size of M-A phases and (iii) significantly more phase fields of M-A. Niobium steel has showed poorest toughness data. Therefore dominating factor in determining toughness of HAZ has been M-A phase fields.

#### **CONCLUSIONS**

Microalloying elements in steels have produced precipitation strengthening by pinning down the grain boundaries for further coarsening. At reheat temperatures before forging, high temperature forming precipitates (e.g. AlN, TiN) have appeared, while at intermediate and low temperatures (e.g. warm working and heat treatments) low temperature forming precipitates

(NbN, V(C,N)) have produced these effects respectively. The improvement in properties have been (i) increase in strength, (ii) resistance to cleavage fracture during impact loading, (iii) refinement of grain sizes, i.e. ferrite and pearlite sizes in as finished products. Warm forging at a constant velocity has produced warm ductility enough for deformation of products with about 50 % of the cost. This economy has been possible by elimination of many expensive steps required for conventional way of fabrication. Precipitation of carbide forming elements has beneficial effects to rise tempering temperature without appreciable softening. Increasing alloy elements in steels have showed anomalous behaviour than carbon steels, where dissolution rate of carbides have delayed the growth rate in tempering than Ostwald ripening effects. Tests have done on repetitive precipitation - dissolution mechanisms of vanadium and niobium nitrides during transformation of tempered martensite to ferrite. Tempered martensite embrittlement has been catastrophic consequences that arise from lower temperature of tempering. The result of fracture formation has stated to be interplay of carbides with impurities across interfaces at lower temperature. Heat treatment of martensitic stainless steel in another incidence has referred to martensite lath boundary precipitation. Studies on welding has referred to that heat affect by more than one pass to the grain coarsened heat affected zone, within critical temperatures of steel transformation which has lowest toughness from blisters of martensite-austenite zones.

## REFERENCES

- [1] C.J. Van Tyne, D.K. Matlock and J.G. Speer, Microalloyed forging steels, Advanced steel processing and products research center, Colorado School of Mines, Golden, CO 80401 USA, pp. 189-197.
- [2] George Krauss, Vanadium microalloyed forging steels, Colorado school of mines, Metallurgical consultant, Evergreen, Colorado 80439, USA, pp. 1-15.
- [3] M.P. Phaniraj and A.K. Lahiri, (2004), Constitutive model for vanadium microalloyed steel under hot working conditions, *Mater. Sci. & Technol.*, **20**, pp. 1151-1157.
- [4] F. Penalba, M. Carsi, C. Garcia De Andres, F. Zapirain and P. De Andres, (1992), Characteristics of vanadium and titanium microalloyed steels forged at intermediate (warm) temperatures through simulation by torsion, *ISIJ Int.*, **32**(2), pp. 232-240.
- [5] J.G. Lenard and M. Tajima, (1995), Thermo-mechanical treatment of a high Nb-high V bearing microalloyed steel, *ISIJ Int.*, **35**(12), pp. 1509-1517.
- [6] P.L. Mangonon, (1981), The heat treatment of vanadium modified alloy steels. *J. of Metals*, pp. 24-30.
- [7] Aleksandar Todoc, Dejan Clkara, Tomislav Todoc and Ivica Camagic, (2011), Influence of vanadium on mechanical characteristics of air-hardening steels, *FME Transactions*, **39**, pp. 49 - 54.
- [8] Hyung-Tes Park, Soo Yeon Han, Dong Hyuk Shin, Young-kook Lee, Kyung Jong Lee and Hyung Sub Lee, (2004), Effect of heat treatment on microstructures and tensile properties of ultrafine grained C-Mn steel containing 0.34mass% V, *ISIJ Int.*, **44**(6), pp. 1057-1062.
- [9] Tempering processes / technology, Heat Treater's guide, (1995), Practices and procedures for irons & steel, ASM International, pp. 96-110.
- [10] Manabu Tamura, Yusaku Haruguchi, Masahiro Yamashita, Yoshikazu Nagaoka, Kensuke Ohinata, Kohtarou Ohnishi, Eiki Itoh, Hiroyuki Ito, Kei Shinozuka and Hisao Esaka, (2006), Tempering behavior of 9%Cr - 1%Mo - 0.2%V steel, *ISIJ Int.*, **46**(11), 1693-1702.
- [11] Manabu Tamura, Takahiro Iida, Hiroyasu Kusuyama, Kei Shinozuka and Hisao Esaka, (2004), Re-dissolution of VN during tempering in high chromium heat resistant martensitic steel, *ISIJ Int.*, **44**(1), pp. 153-161.
- [12] Jae-Kyung, Byong-Whi Lee and H.C. Kim, (1994), Micromagnetic nondestructive evaluation of isochronally tempered 12% CrMoV steel, *Scripta Met. et Mater.*, **30**, 313-318.
- [13] M. Lambrigger, (2001), Geometry dependent ductile to brittle transition of 10CrMoNbV ferritic - martensitic steel Manet II in terms of critical crack sizes, *Mater. Sci. and Technol.*, **17**, pp. 715-720.



- [14] Se-Hwan Chi and Jun-Hwa Hong, In-Sup Kim, (1994), Evaluation of irradiation effects of 16 MeV proton-irradiated 12Cr-1MoV steel by small punch (SP) tests, *Scripta Met. et Mater.*, **30**(12), pp. 1521-1525.
- [15] Pavol Zahumensky, Jozef Janovec and Juraj Blach, (1994), Some aspects of tempered martensite embrittlement in 3Cr-Mo-V steel, *ISIJ Inter.*, **34**(6), pp. 536-540.
- [16] A. Strang and V. Vodarek:, (1996), Z phase formation in martensitic 12CrMoVNb steel, *Mater. Sci. and Technol.*, **12**, pp. 552-556.
- [17] V. Vodarek and A. Strang, (2000), Compositional changes in minor phases present in 12CrMoVNb steels during thermal exposure at 550°C and 600°C, *Mater. Sci. and Technol.*, **16**, pp. 1207-1213.
- [18] Ghiya S.P., Bhatt D.V. and Rao R.V., (2009), Stress relief cracking in advanced steel material-overview, [Proc. Conf.] Proceedings of the world congress on engineering, **2**.
- [19] Reheating cracking, technical sheets, ewf/iab. www.ewf.be-copyright 2007-instituto superior tecnico.
- [20] MA Dang-shen, CHI Hong-xiao, ZHOU Jian, YONG Qi-long, (2012), Microstructure and mechanical properties of martensitic stainless steel 6Cr15MoV, *J. of Iron and Steel, Research, International*, **19**(3), pp. 56-61.
- [21] R. Paton, (1997), Performance of vanadium bearing ferritic stainless steels, *Iron making and Steel making*, **24**(6), pp. 441-446.
- [22] Karl Kusssmaul, Wolfgang Gaudig and Siegfried Haas, (1991), Metallographic examination of the heat-affected zone of a test weld in a type X20CrMoV 12 1 steel, *Mater. Technol., Steel Research*, **62**(2), pp. 83-85.
- [23] Y. Li, N. Crowther, M.J. W.Green, P.S. Mitchell and T.N. Baker, (2001), The effect of vanadium and niobium on the properties and microstructure of the intercritically reheated coarse grained heat affected zone in low carbon microalloyed steels, *ISIJ International*, **41**(1), pp. 46.