On the Effect of y-phase transformation kinetics upon microstructure response of Cold Heading Quality Steel

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Abstract

Cold heading quality CHQ steel is a versatile form over other steels as they are used non-heat treated; their strengthening mechanism is achieved through cold heading operations. Metal is therefore stretched without applying any source of heat, metal flow during the cold heading operation must depend on grains flow which increases the mechanical properties such as strength, resistance to indentation and toughness. It is therefore necessary to form the isotropic grains before applying cold heading operation may increase the properties. Gamma phase formation during the heat treatment is crucial factor for cold operation. An effort is made in this research work to study and find out the Austenite nucleation and growth morphology of commercial CHQ steel through continuous heating experiments by utilizing lead-bath up-quenching technique at different austenizing temperature ranges. High class Optical Microscope Olympus GX51, scanning electron microscopy techniques have been utilized to reveal and interpret the microstructure and it was found that At the 740°C, the microstructure shows the lack of homogeneity in the structure hence cold-head-ability of CHQ steel is anisotropic but at the high temperature in austenite domain at 60sec the resultant austenite is highly homogenous due to high volume fraction of austenite has been formed then the cold-head-ability properties of CHQ steel turned to be isotropic.

Key words: Up-quenching, cold heading steel, gamma transformation, heating rate etc.

Introduction

Cold heading quality steels are high class family of non-heat treated steels used for manufacturing of small components of automobiles, ships and aerospace vehicles, thermal plants, the parts such as fastener, pinions, connecting rods, spline sockets bolt and nuts are commonly by cold heading techniques. In cold heading operation mechanical properties of such components are indirectly controlled by metallurgical aspects such as heat treatment. The increasing trend towards cold working process produces high production rate which minimizes the overall cost, smooth surface finish with dimensional accuracy but the only drawback is the inhomogeneity in microstructure or abnormal grain growth during the cold heading operation. Therefore it is unavoidable to design such parameters in heat treatment which refines grain size, increases hardness and mechanically stabilize the microstructure by phase transformation. Rapid austenizing (up-quenching) refines the microstructure, eliminate distortion and provide uniform dispersion of carbide particles in a matrix of martensite. Uniformity in thermal treatment and temperature during upquenching cycle produces distribution of isotropic properties in almost every section of the steel component these process parameters induce high strength and grains become in normal state. From a various studies on CHQ steel none of anyone has studied the austenite phase transformation using up-quenching technique. The austenite formation in CHQ steel has a limited attention by steel researchers. By applying process parameters correctly a higher strength can be achieved after cold heading [1-6]. There is no any investigation has been reported so far on gamma formation behavior in Micro-alloyed CHQ steel using up-quenching

lead bath. A precisely attention has been given in this work to study the austenite formation in CHQ steel; this technique can be employed to other grades as well. The distribution of uniform volume percent of austenite in this steel is mechanically stabilized. Due to uniform distribution of microstructural constituents there will be a minute probability of abnormal grain growth during or after cold heading operations as a result steel becomes more favorable for cold heading **[7-8].**

Experimental Procedure

The experimental CHQ steel was obtained through the process as shown in Fig 1.



Figure 1. CHQ steel manufacturing process.

The chemical composition of the steel is shown in Table 1.



С	Si	Mn	Ν
0.463	0.241	0.872	0.0049

Table1. Chemical composition (Wt%) of the experimental steel.

Experimental steel was heated up to 1200°C and was hold at that temp: for 120 min for solution treatment. After solution process the steel was engaged in hot rolling operation to reduce its thickness. The 80% reduction was achieved in 4 - passes. Steel plates were then cut at 800°C, after cutting it was air-cooled. Specimen cutting was performed to reduce the size of the plates. Using lead-bath furnace connected with thermocouple following cycle was employed. Fig2.



Figure 2. Rapid heating cycle in lead bath up-quenching.

Austenizing temp: for up-quenching heat treatment was selected by ASM metals hand book. [8]. After austenization the samples were water quenched and were cut perpendicular to rolling plane direction and were mounted and polished according to standard metallographic methods followed by polishing step in 1µm and 0.5µm diamond paste as a last step for observing the structural features under photomicroscope. The samples were optical then ultrasonically cleaned in an ultrasonic cleaner in order to remove stubborn contaminants, chemical residual, dirt or any finger print formed on the surface of sample during polishing. Two step etching technique i-e, 2% Nital and 4% Picric Acid was used to reveal the microstructure. The continuous heating experiments were performed in the lead bath as shown in Table.2

No.	Temperature	Time
1	740°C	10, 15, 30, 60
2	770°C	10, 15, 30, 60

3	800°C	10, 15, 30, 60

Table2. Continuous heating experiments.

Microstructure Evolution

Microstructure, as shown in Fig3a, was carried out by using only solution of 2% nitric acid diluted in 100ml ethanol, this etchant was successful to reveal the grain boundaries but only drawback was that it was difficult to distinguish between the eutectoid-ferrite and martensite because both appeared as white, to differentiate between military transformation such as martensite phase and ferrite phase the 2-step etching, 2% nital followed by 4% picric acid was introduced to ascertain between two phases, this microstructure Fig3b. is shown in



licrostructures of up-quenched Base ST at 740C for (a)10sec, (b) 15sec, (c) 30sec, (d) 60sec

Figure 3a: Microstructure using 2% Nital



Figure 3b: micro structure using LePera.

Examination of this microstructure under light microscopy was not again satisfied because still it was unclear to distinct of three phases like ferrite, pearlite and martensite. The color etching technique using LePera solution then was implied to steel samples as described in reference [9]. A detailed inspection under polarized light rather than halogen light in the microscopy successfully discloses three phases individually then it was easy to determine the volume fraction of austenite with respect to increasing time and temperature. Blue, brown off-white and white colors appeared as ferrite, bainite, martensite and retained austenite respectively. Further details about this tint etching technique of LePera can be found in reference [9]. The volume fraction of displacive phase of martensite was calculated by using point counting method as described by R. L. Higginson and C.M. Sellars detailed method can be found in reference [10]. As rolled microstructural features at T_0 , as rolled microstructure shown in Fig4 is a reconstructive phase having ferrite and pearlite only. The territory of pearlite is decorated with allotromorphic ferrite ring commonly termed as pro-eutectoid ferrite, interface between this ring and pearlite colony acts as the austenite grain boundary during transformation. The formation of martensite can take place within this territory because the coordinated motion of military transformation is weak against this ring and cannot cross this grain boundary barrier which behaves as a strong defect. The microstructure obtained at 710°C, 10, 30, 300 sec holding time during up-quenching is slightly lower than eutectoid temperature which shows formation of larger fraction of "new" ferrite phase volume with respect to increasing holding time



Figure 4: Reconstructive phase.

At the holding time of 300sec it can be noticed that deformed grains which are relatively enlarged replaced by a freshly formed bunch of un deformed ferrite grains which are nearly equiaxed that will nucleate and can grow above the eutectoid temperature which can be named as lower critical temperature or AC_1 , this recrystallization can take place until the original ferrite grains have been absolutely ingest as shown at 300 sec holding time. Whenever recrystallization of ferrite grains reaches at higher volume then it is indication of decreasing hardness as well as

strength simultaneously, but ductility may increase on other hand. Spheroidizing provides the needed ductility for cold heading [11] During cold heading operations there is strong driving force for these soft metals to become hard by cold working phenomenon consequently they may lose their softening property and further there is a strong driving force of abnormal grain growth of grains and hence microstructure become un-identical In order to decrease the degree of this non-uniformity steel samples were up-quenched at different austenitic domains in lead bath. Microstructural features at T₁ (740°C, 10, 15, 30, 60 sec) After heating at 740°C for scheduled soaking time, the stable recrystallized ferrite become unstable and starts to initiate the nucleation of austenite which moves heterogeneously upon cooling and transformation turned the civilian into military transformation. The extent to which recrystallization completes before the austenite begins to form influences the kinetics of austenite formation and the spread of austenite nuclei in pearlite domain [12]. The insoluble and discarded carbon by austenite and ferrite appears as iron carbide moves at the austenite grain boundaries in the form of spurn carbon which can stimulate the austenite nucleation, was not observed, possible reason is the high heating rates in the upquenching bath so that's why the majority of austenite formation in this steel has obtained only in pearlite territory, due to shorter path it was easy for cementite plate to supply carbon to neighboring ferrite. Evidence of phase transformation can be seen at 30sec and 60sec holding time but the microstructure obtained after 15 and 30 sec holding, identification of austenite phase was not easy. Microstructural features at T₂ (770°C, 10, 15, 30, 60 sec) captured at 770°C can be seen in fig5. The austenite nucleation and growth were observed during entire process but at10sec and 15sec there was a low volume fraction of austenite was observed the possible reason behind this delayed transformation was that up to 10sec holding time there was an incubation period of austenite nucleation however extension in soaking time from 30 and 60sec respectively austenite growth was rapid by consuming pearlite phase, with increasing time degree of ferrite packet size has declined as compare to 740°C microstructure.



Figure 5: Austenite nucleation and growth.

At this temperature it was noticed that the high fraction of pearlite was consumed for mass production of austenite. Microstructural features at T_3 (800°C, 10, 15, 30, 60 sec) reveals that the spiral of

allotromorhic ferrite which was tightly surrounded at lower temperature has been broken and slowly decreasing and almost disappear after 60sec. The stability of austenite growth becomes stronger thus degree of isotropy and homogeneity increases. The response during forming of Austenite is much different both at low and high temp: At elevated temp: into the gamma region, the degree of transformation first increased and then decreased because at high temperature difference in free energy of the parent phase and product phase drastically declined however the diffusion coefficient raised at higher level, it means minor phase has become major phase now and austenite phase has been stabilized. To oversee this logical phase difference at low temperature i-e 740°C and high temperature i-e 800°C, SEM micrograph has been obtained as can be seen in Fig6.



Figure 6: SEM micrographs.

Austenite forming increases the hardness and subsequent tempering can reduce the hardness and improve the toughness so both processes simultaneously can be achieved through up-quenching in lead bath. Aus-forming results in instantaneous increases in hardness value, but this effect is substantially reduced after tempering under conditions which are suitable for fasteners [13].To stabilize the gamma thermodynamically, some alloying elements like nickel and manganese are added in the steel, shape deformation on the other hand mechanically stabilize the structure, this shape deformation involves movement of glissile interface [14]. To ensure that either this movement becomes phase change (displacive) or just becomes reorientation of the lattice

(reconstructive) SEM micrographs at early stage 740°C, 60sec and final stage 800°C, 60sec has been obtained. It is observed that the displcaive transformation grows to a limited size at 740°C in which ferrite plate transforms slow but at final stage at 800°C transformation propagates rapidly by forming a martensite plate which has very high strain energy against the dislocations which were formed during shape deformation of austenite in invariant plane strain. The mechanical stabilization occurs when strain required to initiate the stabilization should be higher than the opposing dislocation which has a glissile interface that has to cross the dislocation barrier. Further details can be found in [15-16]. Interface is nothing but the set of dislocations which allow the two crystals to connect each other during transformation this sort of interface is known as glissile interface which must exist between parent and product phase that allow rapid transformation without any diffusion involving in the system. Displcaive transformation is only possible when such an interface between two crystals can be created. It can also be observed from these figures that an invariant line has left behind between the product and parent phase that means there is no any distortion or rotation along this line and hence the atomic arrangement of austenite and martensite can match perfectly at that line and transformation that changes the parent phase, austenite into the product phase, martensite, leaves at least one line undistorted and un rotated which is evidence that the transformation is displacive rather reconstructive. A detailed investigation of microstructural features at each temperature 740 to 800 respectively indicates that even all the pearlite is digested on forming of austenite but a small trace of the boundary line is still left behind which is nothing but the p-austenite grain boundary so the gliding of glissile interface is possible within this protected area since glide of atoms cannot be endorsed across these austenite grain boundaries. Hence it can be concluded that grain boundaries in the microstructure, presence of dislocations, available interstitial atoms, stacking faults and vacant lattice site are normally not essential to describe an equilibrium state of phases, however if they present in the materials they can promote phase transformation [17-20].

Results and Discussion

In the light of above experimental and theoretical background it is worth to share that up-quenching refines the grain size so there is strong tendency finer microstructure can be achieved in conventional CHQ steels without additional alloying and heat treatments. Uniform distribution of microstructure can increase the rate of elongation thus cold-heading of CHQ steel can be enhanced. The rate of nucleation and rate of growth increases at higher temperatures to form the austenite phase in bulk. Rapid heating (Up-quenching) is the main cause which could not permit to carbides at ferrite-ferrite interface, if they were present before phase transformation, to diffuse to rich in carbon that could be austenite and martensite upon cooling. With increasing holding time and temperature pearlite packet size decreases and consecutively turned into martensite upon cooling.

Conclusion

Three stages during austenite formation in up-quenching of a plastically deformed CHQ steel of 80% reduction in thickness of steel plate have been studied at different holding temperatures (710°C, 740°C, 770°C and 800°C). It has been found that microstructural features and mechanical stabilization of plastically deformed steel specimens can be re-stored to their normal pre-deformed states using up-quenching treatment during this process recovery, recrystallization and grain growth phenomenon has occurred. Gamma formation is possible in pearlite phase and there is no evidence found the nucleation of austenite at ferrite-ferrite grain interface.

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REFERENCES

1. T. Das, "Evaluation of two AISI 4037 cold heading quality steel wires for improved tool life and product quality" Materials Engineering and Performance, vol. 11: 1, 2002, pp. 86-91.

2. Xiaoyu MA "Effect of Microstructure on the Cold Headability of a Medium Carbon Steel". ISIJ International, vol. 44:5, 2004, pp. 905-913.

3. J. Huang. "Austenite formation during intercritical annealing"Met and Mat trans A, vol. 35:11, 2004, pp 3363-3375.

4. R. Hill: "Annealing: The First Step in Cold Forming," *Wire J. Int.*, 2005, *17*(7), pp. 95–97.

5. A. Sabih, "Internal ductile failure mechanisms in steel cold heading process" ISIJ International, vol. 45:8, 2005, pp.1179.

6. Savran, "Microstructural features of austenite formation in C35 and C45 alloys" Materials Transactions A, vol. 38. 2007, pp. 946.

7. Jari Rämö"Influence of strain rate and adiabatic heating on the deformation behavior of cold heading steels" Materials Processing Technology. Vol. 209:11, 2009, pp 5186-5194.

8. J. R. Davis, et al., "Annealing of steel," Heat treating, vol. 4, ASM International, 2011.

9. Metallographic Methods for Revealing the Multiphase Microstructure of TRIP-Assisted Steels.

10. R.L Higginson, C.M Sellars Worked Examples in Quantitative Metallography.

11.J.W. Christian "Theory of Transformations in Metals and Alloys".2012.

12. A. Sabiha "Internal ductile failure mechanisms in steel cold heading process" Mat Sci vol. 6, 2013. Pp.46

13. Seawoong "stability of ultrafine-grained transformationinduced plasticity steel with Mn partitioning". ISISJ International, vol. 65. 2013, pp. 634-640

14. H. K. D. H. Bhadeshia. "Mechanical stabilisation of austenite" Materials Science and Technology, vol. 22, 2013, pp.641.

15. Xueda Li "Influence of prior austenite grain size on martensite–austenite constituent and toughness in the heat affected zone of 700MPa high strength linepipe steel" vol. 616, 2014, pp.141-147.

16. C. Goulas "Microstructure, texture and mechanical properties in a low carbon steel after ultrafast heating"Mat Sci and Eng. A. vol.672, 2016, pp.108-120.

17. F.M.Castro Cerda "Austenite formation in 0.2% C and 0.45% C steels under conventional and ultrafast heating" Materials and Design vol. 116, 2017, pp. 448.

18. AnishKarmakar "Effect of composition and isothermal holding temperature on the precipitation hardening in Vanadium-microalloyed steels". Materials Characterization, vol. 132, 2017, pp. 31-40.

19. Shahid Hussain Abro, "Effect of Heating Rate on Microstructural Developments in Cold Heading Quality Steel used for Automotive Applications" Mehran University Research Journal of Engineering & Technology Vol. 37, 2018, pp.461-466.

20. Bo Wang "Precipitation Behavior of VN in High Nitrogen and Vanadium Micro-alloyed Low Carbon Weathering Steel" Transactions of the Indian Institute of Metals. Vol. 71:70, 2018, pp. 1607.