Study the effect of concentration of ethylene glycol on heat transfer and hardness during quenching of EN9 steel

[1]Bhamare Devendra M, [2]Sachin V Mutalikdesai, [3]G P Bharambe
Department of Mechanical Engineering, Trinity College of Engineering and research, Pisoli, Pune.

***Abstract* – Heat treatment is mainly employed to alter the physical properties of the steels. After heat treatment Steel components are generally quenched in forced gas, oil or water flow to improve mechanical properties and improve product life. During the quenching process, rapid heat transfer takes place from hot metal component to the surrounding quenching medium. This rapid heat transfer in quenching process introduces the temperature gradient as the surface cools faster than core of the heated component. This variation in cooling rates may give rise distortion, cracking and high residual stresses along with variation in hardness. This variation is due to difference in conduction and convection heat transfer rates. To minimize such problems while improving mechanical properties, it needs to optimize the process for both part geometry and quenching process design. In this work the attempt is made about reducing the convective heat transfer to avoid the distortion and cracking by addition of ethylene glycol in water with different concentrations. The EN 9 steel material model behavior during quenching is to be observed and study the heat transfer characteristics along with changes in properties of steel specimen. The data obtained by experimental work is be plotted to observe the effect on cooling rate and the material property like hardness is measured using Rockwell hardness tester with cone shape diamond indenter.**

***Index Terms* – Quenching, distortion, cracking, residual stress, ethylene glycol, temperature gradient.**

1. INTRODUCTION

Quenching is the important process of heat treatment which is mainly used to alter the physical properties such as microstructure and mechanical behavior, and sometimes chemical properties such as carbon concentration, of a material or a part. During this process, steel is rapidly quenched from its austenitising temperature typically in the range of 845^0C to 870^0C in surrounding quenching medium. Quenching is the process of heat treatment without material removal. General heat treatment processes include quenching, tempering, aging, annealing, normalizing, etc. and those involving chemical property changes such as carburizing, nitriding, etc. This work studies heat transfer, stress, distortion and material property variation of steel components during quenching in aqueous solution of ethylene glycol. To improve mechanical properties, steel components are usually subject to a heat treatment followed by quenching in different mediums. Quenching is a rapid cooling, which prevents low-temperature processes such as phase transformations from occurring. In this rapid cooling process, heat is transferred out from the hot components to the surrounded cool quenching media. A significant amount of residual stresses can be also developed in the component when quenched particularly in water. The existence of residual stresses, in particular tensile residual stresses, can have a significant detrimental influence on the performance of a structural component. In many cases, the high tensile residual stresses can also result in a severe distortion of the component, and they can even cause cracking during quenching or subsequent manufacturing processes.

In order to prevent the harmful effects of distortion, residual stress and cracking while improve the mechanical properties of steel alloy components, it is highly necessary for heat treaters to optimize component designs and heat treating processes. Experimental trials are used to determine better component designs and process setups, but more and more attentions are being paid to numerical modeling using finite element packages and computational fluid dynamic packages for the benefits on money and time saving. Numerical simulations of quenching of metal parts are usually carried out by finite element analysis packages such as ABAQUS, ANSYS, etc., a CAD model of the part needs to create in 2-D or 3-D form, then the model can be meshed with suitable elements. For quenching simulations, the temperature-displacement simulation is usually decoupled to thermal simulation and structure simulation in industrial practice for two reasons. First, decoupled simulation scheme requires less memory and converges faster than the coupled one. Second, the results from these two schemes are similar since in heat treating processes the heat generated by deformation is usually negligible compared to the heat transferred from hot solid to environmental media. Thus, thermal simulation needs to be first carry out to obtain temperature-time profile of the part. The followed structural simulation reads the temperature-time profile and predicts quenching results such as distortion and residual stresses. In order to obtain high accurate simulation results, the finite element modeling must be validated by experimental measurements of residual stresses, distortion, etc.

A. Buczek and T. Telejko [2004] studied the inverse determination of the conditions during boiling water heat transfer while quenching. They present the technique to determine the values of local heat transfer coefficient. A dynamic method was applied, using inverse solution of heat transfer equation. In this solution the experimentally measured temperature is set in the equation. They finally conclude that due to intense heat transfer, clear temperature gradients are achieved close to the surface being cooled down, which is advantageous to the result of inverse solution. [1]

Li Huiping, Zhao Guoqun, Mu Yue and He Lianfang [2006] studied the quenching process for determination of heat transfer coefficient. They had conducted experiments and used the inverse heat conduction approach for determination of HTC. The authors introduces a new method to calculate the temperature-dependent surface heat transfer coefficient during quenching Process and calculated the surface heat transfer coefficient according to the temperature curve gained by experiment. They stated that during the calculation process, the phase-transformation volume and phase-transformation latent heat of every element in every time interval can be calculated easily by FEM. The temperature and Phase-transformation volume of every element are calculated with the coupling calculation of phase-transformation latent heat. From the literature it is clear that from temperature curves, the temperature-dependent surface heat transfer coefficients of inverse heat conduction problem with the phase-transformation latent heat are evaluated using FEM and the improved advance–retreat method and the golden section method. Also during the process of calculation, the phase transformation latent heat is coupled with temperature and phase-transformation. The heat transfer coefficients gained using FEM and optimization method are compared with the results of reference. It shows that the precision of the method given in this paper is satisfactory, and the convergence speed of iteration is very rapid. The temperature curves are obtained using FEM software and according to these temperature curves, the Temperature dependent surface heat transfer coefficients can be evaluated. [2]

The evaluation of surface heat transfer coefficients [2007] by using experiment measurement method is carried out . According to the characteristics of quenching process, a high-speed data acquisition system for measuring The temperature variations in a quenched part is needs to be set up by using industry standard architecture (ISA) which is discussed in this paper. Cooling curves of P20 steel quenched in 20 ^{0}C and 60 ^{0}C water were acquired by using this system. [3]

Peter Fernandez and K Narayanprabhu [2006] made an attempt to determine the heat flux transients during quenching of Ø28mm×56mm height and Ø44mm×88mmheight AISI 1040 steel specimens during lateral quenching in brine, water, palm oil and mineral oil and the heat flux transients are Estimated by inverse modeling of heat conduction. The variation of heat flux transients with surface temperature for different quenching media was investigated in different experiments. Higher peak heat flux transients are obtained for 28mm diameter specimen than 44mm diameter specimen during quenching in aqueous medium. The study leads to the final conclusion that agitation of quenching medium increases the peak heat flux during the quenching of steel specimen in all the quenching mediums. Peak hardness is obtained at the surface and with smaller diameter specimens during Agitation. The outcomes of their study can be summarized as

1. Agitation of quenching medium increases the peak heat flux during quenching of steel specimen in all the quenching mediums.
2. The time required to obtain the peak heat transfer rate increases with increase in diameter.
3. Higher peak heat flux transients are obtained for 28mm diameter specimen than 44mm diameter specimen during quenching in aqueous medium.
4. Nucleate boiling stage is delayed in 44mm diameter specimen compared to 28mm diameter specimen.
5. Peak heat transfer occurred at lower surface temperature with larger diameter specimens and more severe quenching media. [4]

Bowang Xiao, Qigui Wang, Parag Jadhav and Keyu Li [2010] studied the influence of quenching orientation and agitation conditions on heat transfer of aluminum alloys during water quenching. It has been observed that the quenching process consists of film boiling, nucleate boiling and convection heat transfer. Highest value of HTC is observed during nucleate boiling and convective heat transfer gives the lowest value for the same. Also HTC varies with the orientation of the object in the quenching medium regardless whether the water is agitated or not. The experimental results gives the fact that Agitation enhances heat transfer process especially when objects are at high temperatures and heat transfer process is in the film boiling stage. The analysis of Heat transfer in water quenching of cast aluminum alloys had been experimentally investigated by the authors under various quenching conditions. Conclusions of this work can summarized as: 1. Heat transfer in water quenching undergoes three main stages, namely film boiling, nucleate boiling and convective heat transfer

2. The nucleate Boiling gives highest heat transfer coefficients And the lowest HTC is observed in the convective Heat transfer.
3. Quenching orientation affects heat transfer.
4. Agitation enhances the heat transfer process when Objects are at high temperatures and heat transfer process is in the film boiling stage. [5]

Kermanpur [2010] had verified the Application of polymeric quenchant in heat treatment of crack-sensitive steel Mechanical parts. During their work, the quenching process of the automobile tie rods in different media including water, oil, and a polymeric solution were used and the microstructures and mechanical properties of the rods were predicted by a finite element simulation model. Considering the results of the simulations and the experiments, the optimum Quenchant was selected as Poly Alkaline Glycol (PAG) solution and the tie rods were heat treated using PAG. The results Showed that the use of PAG gave the better results as compared to water and oil quenchants. The distortion and cracking was also reduced by considerable amount as compared to water quenching with improved mechanical properties which were not achievable by oil quenching. From the simulation and experimental results it can be concluded that - Water quenching can be used for hardening AISI 1045 Steel parts and it results in desirable microstructure and mechanical properties. Also the main drawback of using water is the considerable thermal gradients and the volume changes during the martensitic phase transformation, gives rise the high residual stresses, distortion and cracking. - Polymeric aqueous solutions are able to provide a range of cooling rates between water and oil. Also when 10% PAG concentration solution is used it outperforms the other quenchants by giving the better microstructure and mechanical properties with less distortion . [6]

Hengliang Zhang [2010] Studied the Cooling of steels after the high temperature forming process and impact of the heat transfer on the metallurgical structure and the mechanical properties of the part. From the study it had been cleared that the rate of heat removal from a heated component by a quenchant depends on the ability of the liquid medium to wet and spread on the surface from where heat needs to be removed. Generally Quench hardening is a process which is used to produce steel components with reliable service properties such as high strength, hardness and wear resistance. During quenching of steels Distortion, cracking, distribution of microstructure and residual Stresses the most common problems. [7]

Ashok Kumar [2010] studied the Sensitivity of material properties on distortion and residual stresses during metal quenching processes to investigates the effect of thermal, metallurgical and mechanical properties on the final distortion and residual stresses during metal quenching processes. They use the Finite Element Method (FEM) to solve the coupled partial differential equations while doing this the effects like phase transformation enthalpy, transformation-induced plasticity and dissipation were considered. The curvature and the volume averaged effective stresses were considered for the measurement of distortion and residual stresses, respectively. The sensitivity of the density, specific heat capacity, thermal conductivity, transformation start and end times, martensitic transformation coefficient, martensite start temperature, bulk modulus, shear modulus, yield strength and hardening modulus were the main concern in this work. It is found that reduced metallurgical properties, yield stress, and bulk modulus simultaneously lower the distortion and residual stresses for an equal cooling. [8]

2. EXPERIMENTAL WORK

2.1 Assumptions

The medium carbon steel specimen taken for the experimental work as well as quenching medium are considered homogeneous. Properties of quenching medium changes with respect to temperature. Changes in Latent heat during phase change solid – solid of specimen material is neglected. Domain boundaries are considered to be continuously expanding and hence heating of medium due to boundary is neglected. Initially fluid is considered at rest i.e. no convection at start of experimental trial. No agitation is provided to specimen. Temperature at start of trial is uniform for quenching medium as well as for solid specimen.

2.2 Material composition

Table 1: Material properties of en 09 steel

No	Constituent	% content
1	Carbon	55 %
2	Manganese	09 %
3	Phosphorous	0.04%
4	Sulphur	0.05%
5	Iron	Remaining

The specimen model is cylindrical roller with diameter 0.05m and length 0.1m.

2.3 Specimen boundary conditions:

At t =0 sec Ts = 1173 k Quenching medium initial temperature: 298k

Fluid domain size: 30cm X 30cm X 20cm

Fluid domain Boundary Condition: Tm= 298k, P = 1.013 bar

2.4 Heat Treatment of Specimens

The specimen are made from EN 9 steel rod with diameter 0.05m and length 0.1m. Additional specimens are also made for measuring the hardness before and after Quenching. All specimens are heated in muffle furnace up to 900°C for 15 minutes and then transferred in the aqueous solution of ethylene glycol with different concentrations i.e. water, 20%, 40% and 60% of EG in water by mass. The temperature range and hardening/tempering soaking times for the experimental investigations were selected based on the material composition of the specimens. The K type thermocouples are brazed at the center and surface of the specimen to measure temperature variation. Quenching process is followed by grinding and then polishing. Next phase is etching by using mixture of nitric acid and methanol and then the microstructures of the specimens before and after heat treatment have also been observed using microscope.

2.5 Equipment used

1 Muffle furnace
2 Quenching tank
3 Steel specimen
4 Thermocouples
5 Microscope
6 Rockwell hardness tester
7 Pliers
8 Temperature indicator

Fig 1. Experimental set up

3. RESULTS AND DISCUSSION

3.1 Observations: The data obtained from experimentation is tabulated below

Table 2: Temp vs. Time readings for 0% EG

Time	Temp		Time	Temp	
	Centre	Surface		Centre	Surface
0	900	900	140	100	70
5	870	650	160	85	60
10	840	450	180	70	55
15	625	400	200	60	50
20	600	315	220	54	46
40	450	230	240	50	43
60	300	175	260	48	35
80	200	115	280	40	33
100	180	100	300	32	31
120	130	80			

Table 3: Temp vs. Time readings for 20% EG

Time	Temp		Time	Temp	
	Centre	Surface		Centre	Surface
0	900	900	140	105	70
5	880	660	160	90	65
10	850	460	180	83	60
15	780	410	200	75	56
20	740	350	220	60	52
40	550	250	240	55	45
60	420	190	260	50	38
80	270	150	280	40	35
100	155	100	300	34	30
120	125	80			

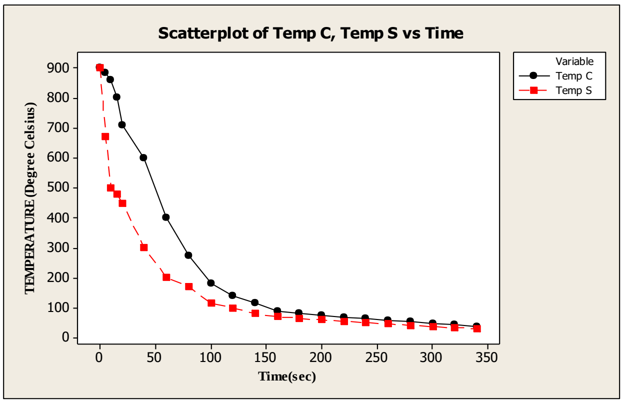
Table 4: Temp vs. Time readings for 40% EG

Time	Temp		Time	Temp	
	Centre	Surface		Centre	Surface
0	900	900	160	90	70
5	885	670	180	80	65
10	860	500	200	75	60
15	800	480	220	68	55
20	710	450	240	63	50
40	600	300	260	59	46
60	400	200	280	54	41
80	275	170	300	48	36
100	180	115	320	43	33
120	140	100	340	36	31
140	115	80			

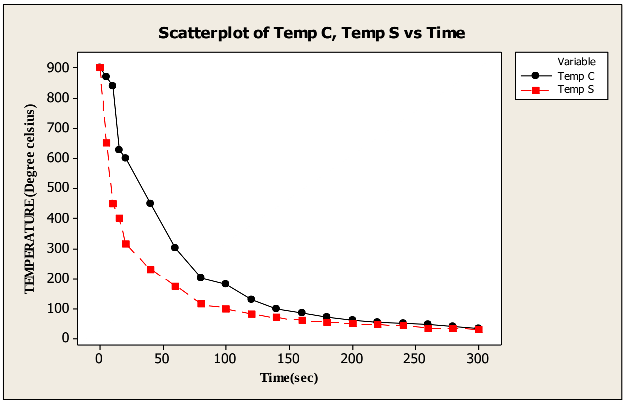
Table 5: Temp vs. Time readings for 60% EG

Time	Temp		Time	Temp	
	Centre	Surface		Centre	Surface
0	900	900	160	130	100
5	885	710	180	110	85
10	875	620	200	90	75
15	840	580	220	80	60
20	800	520	240	72	54
40	625	360	260	64	48
60	470	270	280	58	43
80	360	205	300	52	38
100	265	160	320	45	35
120	210	135	340	38	33
140	150	115			

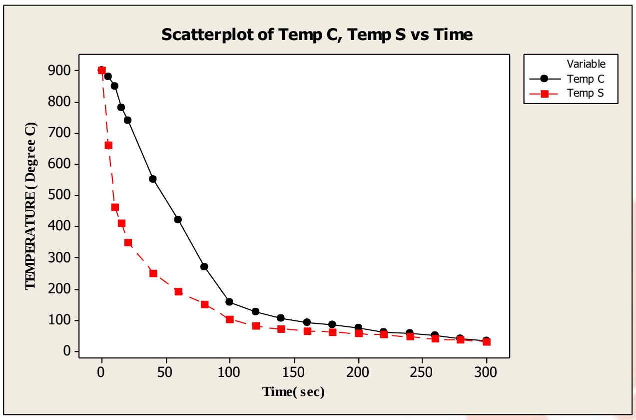
3.2 Temperature vs Time



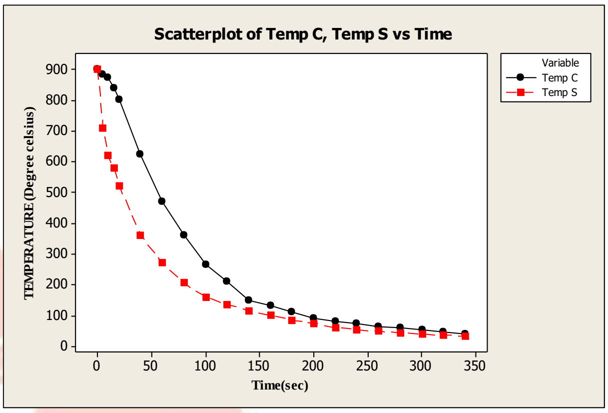
(a) Temperature vs Time Plot for 0 % EG Concentration



(b) Temperature vs Time Plot for 20 % EG Concentration



(c) Temperature vs Time Plot for 40 % EG Concentration



(d) Temperature vs Time Plot for 60 % EG Concentration

The graph (a) shows variation of temperature with respect to time for surface and core. We can observe large temperature difference between surface and core of the specimen. Due to variation in cooling of surface and core there will be uneven contraction of material of specimen which is responsible for residual stresses.

The graph (b) shows variation of temperature with respect to time for surface and core when quenching medium is 20% ethylene glycol solution by mass. The temperature variation between surface and specimen at particular instant is less compare to previous quenching medium which leads to less residual stress formation compare to previous trial.

The graph (d) shows variation of temperature with respect to time for surface and core when quenching medium is 60% ethylene glycol solution by mass. The cooling rate is slowest for this trial. As percentage of ethylene glycol increases in aqueous solution rate of heat transfer from surface to quenching medium decreases. Heat transfer by convection approaches the heat transfer by conduction within specimen and hence temperature gradient between surface and core of specimen is least for this trial. We can predict that residual stress formation is least for this trial. As percentage of ethylene glycol increases in quenching medium the temperature gradient goes on decreases and it will result in less residual stress formation. We can also ensure the formation of martensite throughout the work piece by comparing slowest cooling curve with critical cooling curve.

4. HARDNESS TEST

The experiment is conducted by heating of EN 9 steel specimen followed by quenching it in different concentrations of ethylene glycol in water and the time temperature data obtained is used to lot cooling curves. Cooling curve analysis showed the reducing convective heat transfer rate at the surface of the test specimen. This reduction in cooling rate results in lower hardness values as the concentration of ethylene glycol increases. After experimental trial, hardness of the materials specimen is tested by using Rockwell hardness tester with following specification

Rockwell hardness test
Indenter type: Diamond / Cone
Scale : Black
Load applied : 100 kg.

Table 6: Hardness measurement

EG Concentration	Before quenching		After quenching	
	Surface	Core	Surface	Core
0 %	32	30	61	58
20 % (1.8 lit)	32	30	60	58
40 % (3.6 lit)	32	30	54	52
60 % (5.4 lit)	32	30	51	50

5. CONCLUSION

Based on the observations and the graphs plotted the following concluding remarks are made as a result of variation of quenchant concentration and its effect on EN 9 Steel:

1) There is variation in cooling rate at the surface and core of the heated object and it causes uneven contraction of specimen which results in distortion and may leads to crack formation along with residual stress.
2) Increase in the concentration of ethylene glycol reduces the rate of convective heat transfer from the surface, which reduces temperature variation between surface and core during quenching.
3) Reduction in variation of temperature between surface and core ensures less possibility of distortion and cracking.
4) Due to addition of ethylene glycol Heat transfer coefficient decreases.

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