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Effects of annealing and annealing cooling media in corrosion resistance and microhardness of low content chromium nickel steel

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ABSTRACT

To study the effect of intermediate spheroidal annealing on some properties of a metal, samples of chromium-nickel steal were chosen. The samples were carburized, normalized, annealed, hardened and tempered. The microhardness and corrosion resistance in acidic media were measured. The effect of annealing cooling media was also studied. Cooling media was chosen in a way to characterize the effect of cooling rate on steal properties: furnace cooling media; slow cooling rate, air cooling media; fast cooling rate, and oil cooling media; fast cooling rate. This paper showed that intermediate spheroidal annealing affect in alloys properties. It was found that, the hardness of the material increases and decreases in dependence on annealing temperature and holding time, but the results weren't predictable; each temperature and cooling rate has its pattern. The study shows an increase in the corrosion resistance of the material due mainly to spheroidal annealing process. In general the best result showed samples annealed at 740 °C, 60 min. for microhardness, and 740 °C, 45 min for corrosion resistance, especially for oil cooling media.

KEYWORDS

Corrosion;
Resistance;
Microhardness;
Microstructure;
Weight loss;
Intermediate annealing;
Spheroidal annealing.

INTRODUCTION

One of the heat treatments or thermal processes employed on the iron steel is annealing process, which is usually carried out to relieve stress, and to increase softness, ductility, and toughness[1]. These properties are achieved by refining, uniforming, and/or spheroidizing the grains of pearlite microstructure due to annealing processes[2]. Spheroidal annealing gives maximum softness because of the transformation of cementite lamellae to spheroids. Annealing process usually carried out as the last stage of heat treatment processes.

Recently in many works annealing processes are used for other purposes. For example, rapid annealing used to study the nucleation step in a number of transformations and to control the scale and spatial distribution of a variety of reaction products[3]. Han et. Al. (2008) have improved the strength about 50MPa of pure copper using cyclic equal channel angular pressing with intermediate annealing during pressing[4]. Also different temperatures and periods of intermediate annealing were used in an attempt to improve strength and impact toughness resistance of high martinsite dual phase steel[5]. Intercritical annealing heat treatments for reinforcing steel have illustrated a good corrosion resistance[6] which proves to be good for industry since it always suffers financial loss due to material deterioration which is a loss in materials directly or indirectly. A direct loss in materials is due to the corrosion of a part of equipment that will cause it to stop functioning correctly. This direct loss in materials may cause damage to parts that depends on the direct damaged part. Such as the operation interruption of industrial plants, over dimension of structures and equipments and mainly the losses imposed to the society and the environment due to toxic or inflammable chemical accidents or leaks. The quantity of deteriorated materials varies from 15 to 25% of the steel produced in the world, making the cost of the product reaches 4.0% of the gross product industrially produced.[7]

Gooch (1995)[8] stated that; the failures in metallic components may happen in a very fast way preventing from detecting the problem in time to be repaired, thus causing high costs expenses.

In the early 60's the soft martinsitic stainless steels were developed in Switzerland aiming to improve the mechanic and corrosion resistance[9]. Due to that, it is necessary to study even more the alternatives to lower the maintenance cost of corrosion in such an aggressive environment, and this paper aims to evaluate the behavior of the martinsitic stainless steel mainly Chromium nickel steel alloys.

Chromium nickel steel alloys has good mechanical properties, and these properties are higher in present of martinsitic microstructure. However, these alloys can't be used in aggressive acidic media because of their poor corrosion resistance in such media. For these alloys high corrosion resistance could be achieved by adding many other alloying elements such as molybdenum and nitrogen. The corrosion-resistant of chromium-nickel--molybdenum alloys are often required for general construction of the components and for protective lining of the systems inside many chemical processing factories, where the environment is very corrosive.

To achieve more satisfaction properties for these alloys many heat treatment processes can be used and/or cold working, such as cold equal-channel angular pressing austenitic Cr-Ni steel gives yield strength of 1090MPa relative to the initial with 320MPa[10].

The goal of this work is to study the effect of using annealing process as intermediate stage between other heat treatment processes on microhardness and corrosion resistance of chromium-nickel steel. For this purpose Samples from Chromium-Nickel steel were carburized to increase the concentration of carbon on the surface of the steel. The process of intermediate annealing was added to disperse uniformly the carbides in the steel. This uniform dispersion will affect on some properties of the steel. The temperatures were chosen in the range of annealing processes[11]. The effect of temperature, annealing time, and cooling media was also studied.

EXPERIMENTAL

Materials and heat treatment processes

All specimens of Chromium-Nickel (TABLE 1) steel with a diameter of 8, 12 mm and 100 mm length were carburized in a Liquid carburizing furnace in 30% NaCN for 100 min.

Standard samples were treated in the following sequence: carburized, hardened then tempered. The remaining samples were carburized, normalized, annealed, hardened and tempered in the mentioned sequence refer to TABLE 1 for heat treatment conditions. For annealing process; the annealing temperatures 740, 760, and 780 °C were selected in holding time intervals of 45, 60, and 75 min. the specimens after exposure time were cooled in deferent cooling media. Cooling media was chosen in a way to characterize the effect of cooling rate on steal properties: furnace cooling media; slow cooling rate, air cooling media; fast cooling rate, and oil cooling media; fast cooling rate.

TABLE 1 : Chemical composition in weight percent of Cr-Ni steel

Element	C	Ni	Cr	Mo
Wt %	0.19	3.7	1.3	0.2

Microhardness

Vickers microhardness was conducted using a

Highwood HWDM-3 (TTS Unlimited Inc., Japan) instrument. The micro hardness was measured at three different locations of the specimen, and then the average values were calculated.

Corrosion test

Corrosion studies with chromium-nickel steal alloy were carried out by weigh loss method to investigate the effect of intermediate annealing on corrosion in steel alloy. For this purpose cylindrical steel samples were polished up to 400 grit using polishing papers, accurately weighed (typical dimension of 8mm diameter×20mm), then immersed in 150mL of 0.1M HCl solution for the desired exposure time. After each exposure time the samples were removed from its beakers, dried and re-weighed to a constant weight using an analytical balance with accuracy of ±0.0001g.

RESULTS AND DISCUSSION

Microhardness

The results of Vickers microhardness was recalculated in HRC. The representation of the results is in TABLE 3 in which microhardness of the steel depends on the annealing temperature and holding time for deferent cooling media. The initial microhardness for the carburized sample (standard) is 39.8 HRC. It is obvious that, intermediate annealing affect on microhardness for all samples, even though the effect varies in dependence on annealing temperature, holding time, and cooling media. This effect shows negative and positive deviation at standard. To find optimum annealing parameters for best results the results must

TABLE 3 : The results of microhardness test.

Sample No.	Annealing temperature °C	Annealing time (min)	Microhardness, HRC		
			cooling in furnace C	Cooling in air	Cooling in oil
1		45	34	32.6	57
2	740	60	50.4	52.5	58.8
3		75	38.6	34.53	35
4		45	33.5	53	33
5	760	60	33.9	54.83	31
6		75	30.9	32.4	30.9
7		45	48	51.7	41.8
8	780	60	45.6	50.2	37
9		75	35.3	50.8	35

be analyzed according to annealing temperature, time, and cooling media.

Effect of intermediate annealing at 740 °C

The effect of slow and moderate cooling gave almost the same results and defer from the fast cooling which gave better results. It appears that, the microhardness depends also on annealing holding time. It's shown that the microhardness increases with increasing annealing time until 60 min, where the results for deferent cooling media better than the standard, then reduces for 75 min annealing time and all the results less than the standard. The best results were for 60 min holding time and oil cooling media where the result is 58.8 HRC, which gives an increasing about 47.74% comparing with the standard.

Effect of intermediate annealing at 760 °C

Increasing intermediate annealing temperature by 20°C seriously affect negatively on microhardness. The results of microhardness for slow and fast cooling rate almost the same and less than the standard. Increasing annealing holding time for these cooling rates slightly effect on the results where the results slightly decrease with time.

Effect of intermediate annealing at 780 °C

It was observed that, the results of microhardness for slow and fast cooling media affect on microhardness by the same way, but annealing holding time affect negatively with its increasing. Also the results show good values (the value of microhardness are better than standard) comparing with the standard, but it reduces with increasing annealing holding time.

For moderate cooling rate (in air) it was observed that, the values of microhardness did not change with annealing holding time, and the values of microhardness are the best.

In general the best results were for samples annealed at 740 C, 60 min, especially for samples cooled in oil.

Corrosion test

To determine the effect of spheroidal intermediate annealing on corrosion resistance performance, each steel specimen was immersed in 150 mL, 0.1M HCl solution. The solution was added during the experiments when needed to prevent any reduction in solution concentration. Weight loss was calculated as:

$$W = \frac{m_o - m_i}{A}, \frac{mg}{cm^2}$$

Where:

- M_o is the mass of the specimen before immersing in corrosion media.
- M_i is the mass of the specimen after a period *i* of the immersing time,
- *A* is the surface area of the specimen.

The results of weight loss for each specimen was drown in a chart (see Figure 1 not all the result shown for clarity) and an equation for a trend line was found. All equations were linear; the results of the slop value (dW/dt, $mg.cm^{-2}min^{-1}$) were added to the table with correlation coefficient R^2. The slop for each trend line characterizes the rate of weight loss. The results

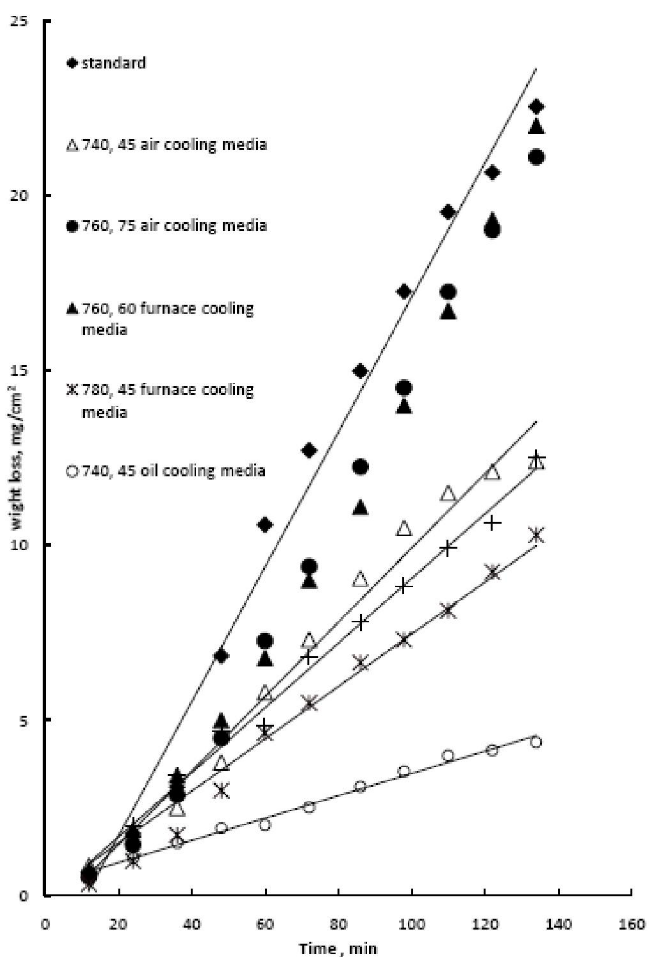

Figure 1 : The effect of exposure time on weight loss for specimens with different annealing temperature, time, and deferent cooling media (legend: first number is annealing temperature, °C, the second is annealing time, min)

are summarized in TABLE 2. An interesting factor was found that, the values of weight loss for all samples were less than the standard, which means that intermediate annealing affect positively on corrosion resistance of the metal independently on the way of cooling the metal after annealing. To find optimum annealing parameters for best results of corrosion resistance, the results of weight loss must be analyzed according to annealing temperature, time, and cooling media.

TABLE 2 : Heat treatment processes conditions.

Condition	Carburizing	Normalizing	Hardening	Tempering
Temperature, °C	930	835	815	180
Holding time, min	100	60	60	120
Cooling media	Air	air	Oil	air

Effect of intermediate annealing at 740 °C

From TABLE 4 one can see that the slops of the equation have the lowest value for specimens cooled by oil (fast cooling) after annealing, where the value less than $0.1mg.cm^{-2}min^{-1}$. The effect of annealing time showed that, the rate of corrosion is increasing with increasing time for fast and slow cooling, and changing with a maximum in 60 min for moderate cooling.

Effect of intermediate annealing at 760 °C

For low and moderate cooling rate it was found that, almost no change in the rate of corrosion with time changing from 45 to 60 min then the corrosion rate increases at 75 min. For fast cooling rate the rate of corrosion did not change with annealing time.

Effect of intermediate annealing at 780 °C

For low cooling rate it was found that, the corrosion rate increases with time with a maximum at 60 min, but for moderate cooling rate it was increasing linearly. For fast cooling rate the rate of corrosion almost did not change with time.

The corrosion resistance in general shows that the best results were for samples annealed at 740 C for 45 min. especially for samples cooled in oil

TABLE 4 : The results of weight loss for samples annealed at deferent temperatures, time, and deferent cooling media

Annealing Temperature, °C	Annealing Time, min	Sample immersing time												
		12	24	36	48	60	72	86	98	110	122	134		
		Weight loss, mg/cm^2												
Standard Sample		0.66	1.75	3.09	6.84	10.59	12.71	14.98	17.25	19.52	20.66	22.54	0.1922	0.9861
Air cooling media														
740	45	0.53	1.66	3.01	4.16	5.32	6.83	7.95	9.25	10.3	11.51	12.22	0.0982	0.998
	60	0.32	1.03	2.36	4.4	6.46	8.9	11.1	12.2	12.92	13.2	13.31	0.1226	0.9441
	75	0.85	1.7	2.51	3.8	5.8	7.3	9.06	10.5	11.5	12.1	12.4	0.106	0.9312
760	45	0.4	1.1	1.98	2.5	3.34	5.5	9.08	11.09	12.45	14.5	17.76	0.1443	0.9565
	60	0.46	1.4	2.92	5	8.59	10	12.31	13.5	15.18	16	17.87	0.1503	0.9849
	75	0.53	1.44	2.87	4.5	7.27	9.4	12.24	14.5	17.24	19	21.1	0.1788	0.992
780	45	0.31	0.87	1.7	3	5.63	7.01	8.81	10.5	13.07	14	15.5	0.134	0.9883
	60	0.56	1.44	2.81	4.01	5.88	7.8	9.11	11.05	12.95	15.07	17.65	0.139	0.9888
	75	0.42	1.57	3.11	4.37	6.28	8.3	10.23	11.87	13.78	16.8	18.98	0.1516	0.9897
Furnace cooling media														
740	45	0.55	1.12	1.85	2.4	3.16	3.8	4.6	5.4	6.19	7	7.56	0.0585	0.9983
	60	0.71	1.87	3.03	5.5	8.2	10	12.82	14.2	15.63	16.4	17.98	0.1514	0.9861
	75	0.52	1.44	2.77	4.8	6.32	8.5	11.21	14.8	17.34	18	18.7	0.1674	0.9808
760	45	0.58	1.73	3.04	4.5	6.25	8.5	11.52	15	18.58	19.26	21	0.1818	0.978
	60	0.71	1.87	3.42	5.1	6.78	9	11.1	14	16.71	19.3	22	0.1766	0.9849
	75	0.71	2.22	4.29	6	8.42	12	15.38	20	23	25.71	27.55	0.2367	0.986
780	45	0.32	0.98	1.73	3.2	4.64	5.5	6.65	7.3	8.13	9.25	10.3	0.0838	0.9935
	60	0.46	1.42	2.81	5.1	7.38	9	11	12.5	13.5	14.2	15.4	0.1308	0.9834
	75	0.4	1.38	2.61	5	6.95	9	11.13	11.8	12.3	12.9	13.56	0.1176	0.9579
Oil cooling media														
740	45	0.6	1.07	1.5	1.92	2.02	2.52	3.11	3.55	4	4.15	4.38	0.0319	0.9908
	60	1.31	2.75	3.12	4.09	4.56	5	5.74	6.97	8.21	8.76	10	0.0664	0.9835
	75	1.09	2.1	2.37	3.17	4.6	6.23	6.88	7.28	7.69	8.51	9.75	0.0707	0.9788
760	45	1.32	2.85	4.2	5	6.2	7	7.41	8.12	8.83	9.95	11.02	0.0727	1.2382
	60	1.02	2.69	2.91	4.09	4.19	4.8	5.26	6.08	6.91	7.23	7.6	0.0504	0.9743
	75	0.63	1.33	2.26	3.19	3.26	3.74	5.04	5.8	6.57	7.23	8.32	0.0607	0.9899
780	45	2.2	3.95	4.27	5.32	5.48	6.42	7.76	8.03	8.31	9.12	10.27	0.0599	0.98
	60	1.34	2.77	3.25	3.85	4.04	4.72	5.72	6.14	6.56	6.95	7.47	0.0471	0.9808
	75	0.59	2	3.42	4.72	4.85	6.81	7.8	8.85	9.91	10.64	12.5	0.0924	0.9924

CONCLUSION

This paper showed how intermediate spheroidal annealing changed some properties of metals. Chromium-nickel steal has been carburized, normalized, annealed, hardened and tempered then compared with standard samples. The cooling media was studied to characterize the effect of cooling rate over steal.

Measurements were taken for the microhardness and corrosion resistance in acidic media and the annealing cooling media to change the cooling rate (furnace, slow, air, fast and oil cooling media).

The study shows that the hardness of the material increases and decreases dependent on annealing temperature and holding time in unpredictable patterns. It also showed fixed patterns for temperature and cooling rate. The study shows an increase in the corrosion resistance of the material due mainly to intermediate annealing process. The best result were for samples annealed at 740C, 60min for microhardness, and 740C, 45min for corrosion resistance especially for oil cooling media.

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REFERENCES

[1] A.Raymond, Higgins; Properties of engineering materials. Hoodder and Stoughton, **(1985)**.
[2] M.H.A.Kempester; 'Materials for Engineers', third edition. Hoodder and Stoughton, **(1984)**.
[3] Florent Lefevre-Schlick, Xiang Wang, David Embury; The production of fine-scale microstructures by rapid annealing Materials Science and Engineering. A, **483–484**, 258–261 **(2008)**.
[4] W.Z.Han, S.D.Wu, S.X.Li, Y.D.Wang; Materials Science and Engineering. A. 14th International Conference on the Strength of Materials., **483-484**, 430-432, 15 June **(2008)**.
[5] K.A.Bello, S.B.Hassan, M.Abdulwahab, U.Shehu, L.E.Umoru, A.Oyetunji, I.Y.Suleiman; Australian Journal of Basic and Applied Sciences, **1(4)**, 407-414 **(2007)**.
[6] Oðuzhan Keleþtemur, Servet Yýldýz; Construction and building materials, **23(1)**, 78-84 **(2009)**.
[7] E.F.Strobel, N.A.Mariano, K.Strobel, M.F.Dionízio; Effect of the Heat Treatment in the Resistance Corrosion of a Martinsitic Stainless Steel CA6NM. 2nd Mercosur Congress on Chemical Engineering. Available online http://www.enpromer2005.eq.ufrj.br/lng/en/index.php.
[8] T.G.Gooch; Heat Treatment of Welding 13%Cr-4%Ni Martensitic Stainless Steel for Sour Service. Welding Research Supplement, 213-222 **(1995)**.
[9] J.D.Crawford, et al.; ASTM STP, **756**, 355-368 **(1982)**.
[10] S.V.Dobatkin, O.V.Rybal'chenko, G.I.Raabb; Acta Materialia, **52**, 1023–1037 **(2004)**.
[11] Mark Andre Maers, Krishan Kumar Chawla; Mechanical behavior of materials. Printice Hall, **(1999)**.