# EVALUATION OF PRECIPITATION HARDENING HEAT TREATMENT OF PH 17-7 STAINLESS STEEL SPRING

**A. A. Babakoohi Ashrafi[1*], H. Mohammadi[2] and A. Habibolla Zade[1]**

\* *alibabakohoi110@gmail.com*

**[1] Metallurgical - Material Engineering Department of Semnan university, Semnan, Iran.**
**[2] Mining and Metallurgical Engineering Department of Amirkabir University, Tehran, Iran.**

**Abstract:**   *In this paper, the influence of heat treatment on PH17-7 stainless steel spring was evaluated. Precipitation hardening phenomenon of PH 17-7 steel was evaluated in three stages. First, the spring constant changes with time and temperature was evaluated. Second, the spring constant changes with respect to its original length at constant temperature and time with blocking (spring length after compression, 18 and 21 mm) were investigated. And finally, the spring heat treatment at 480 °C for 80 min and then holding at 230 °C in oil bath for 60 min without blocking were investigated. The results showed that the use of 18 mm block have large spring constant than 21 mm block. The optimal conditions (480°C for 80 min) for this spring to reaching maximum spring constant were determined.*

**Keywords:** *"PH 17-7 stainless steel, precipitation hardening, spring constant, blocking".*

## 1. INTRODUCTION

Steel 17-7 PH, WNo: 1.4568, is a chromium-nickel-aluminum precipitation hardening stainless steel that classified as high-strength austenite–martensitic stainless steel[1, 2]. Precipitation-hardening (PH) stainless steels are a family of stainless steels that combine high strength, ductility, and good corrosion resistance. This material is widely used as a structural material in a variety of applications in aircraft and spacecraft, and surgical instruments, beds, springs [3,4]. Depending on the thermal and mechanical history, alloys can contain variable amounts of austenite, ferrite, and martensite. The alloy 17-7 PH has approximately the same chromium and nickel contents as austenitic type 301 stainless but also contains 1.2 wt% Al for precipitation hardening. Precipitation hardening in these steels is achieved by homogeneously dispersed fine precipitates formed during aging [5].

The main reason for the development of precipitation- hardening stainless 17-7 PH steel, is the fact that Maragig steels, despite of very high degree of alloying, have not satisfactory corrosion resistance. Therefore, PH steels have been developed. These steels have high values of strength and fracture toughness, very close to Maraging steel, but with greater resistance to corrosion[6].

Strengthening of the steel is achieved by the transformation of austenite into martensite and subsequent precipitation of intermetallic compounds.

The Al-Ni compositions and carbides will precipitate in grain boundary from solid solution phase during hardening heat treatment [7].

After partial or complete transformation austenite into matrensite these steels are additionally strengthened with precipitation aging. This strengthening with precipitation of intermetallic phase NiAl is very simple and effective [8].

Precipitation hardening in these steels is achieved by homogeneously dispersed fine precipitates formed during aging.

PH stainless steels containing Ni and Al can be age-hardened by the formation of NiAl particles. In the mariensitic condition, the aluminum in these steels is in supersaturated solid solution. Upon heating for precipitation hardening, the aluminum in the martensite is precipirated as Ni-Al ($NiAl$ and $Ni_3Al$) intermetallic compounds, as had been identified by electron diffraction studies. The amount of precipitation depends on the time and temperature at which the reaction takes place [9, 10].

The carbon in this steel forms some carbide

*A. A. Babakoohi Ashrafi, H. Mohammadi and A. Habibolla Zade*

such as $M_{23}C_6$, $M_3C_6$, $M_7C_3$. The important carbide is $M_{23}C_6$. When this steel is heated to 1100-1150 °C, the carbides dissolves and will obtain the austenitic phase free of carbide precipitation after quenching. Heating the steel in 480°C–500°C temperature range, $M_{23}C_6$ carbide will precipitate in the grain boundary [11].

Typically the 17-7 PH alloy is furnished is the annealed condition. In this condition, the material possesses an austenitic structure. As an austenitic material, the 17-7 precipitation hardening alloy possesses a relatively low strength. This is the condition in which formability is easiest.

The 17-7 PH Alloy is covered by the AMS 5529, ASTM A313 and EN 10088-3 specification and standards. When 17-7 PH alloy with an austenitic structure is cold worked by substantial deformation, a transformation to martensitic structure results from the deformation. In this condition, the material may be precipitation hardened directly by heat treatment at 482°C. The aim of this paper is to study the influence of effective factors such as temperature and time, blocking and without blocking precipitation hardening on 17-7 PH steel.

## 2. MATERIALS AND METHOD

The chemical composition and dimension of the springs used in the present work is shown are Table 1and 2 [12, 13].

Precipitation hardening of 1.4568 (17-7 PH) steel for obtaining the spring with K (Spring Constant) = 0.19 -0.21 in four stages was performed. The spring diameter in this study is low, so precipitation hardening heat treatment at high temperature (955°C) is prevented, because the high temperature cause to spring deformation and distortion. The melting point of this material is about 1430 °C.

**Table 1.** Steel chemical composition 1.4568 (X7CrNiAL 17-7) for precipitation hardening treatment

| C % | Si % | Mn % | P % | S % | Cr % | Ni % | Al % |
|---|---|---|---|---|---|---|---|
| Max 0.09 | Max 0.7 | Max 1 | Max 0.040 | Max 0.030 | 16-18 | 6.5-7.75 | 0.75-1.5 |

**Table 2.** The characterization of spring used for precipitation hardening

| Description | Attribute (mm) | Description | Attribute (mm) |
|---|---|---|---|
| outside diameter of the spring | 4.65 | Primary length | 36.5 |
| the external diameter of the wire spring | 0.65 | block Length | 18-21 |
| the number of ring diameter coil springs | 18 | Diameter Average spring | 4 |
| the number of useful ring diameter coil springs | 16 | inside diameter coil springs | 3.35 |

Therefore, firstly should obtain an appropriate operating temperature range for precipitation hardening and strengthening. So the precipitation-hardening treatment consisted of heating at 400 °C to 540 °C in a resistance furnace for a period of 35 to 150 min was selected with regarding the condition CH 900 (AMS 5529). The low temperature allows minimum distortion compared to conventional hardening processes.

The samples were arranged inside the reticulated metal basket and then were placed into the furnace with 10°C accuracy and were cooled in air after heat treatment. The final step of this heat treatment cycle is age-hardening, which the specimens were held at 230°C in oil bath for one hour and then cooling, normally very slowly, as shown in Fig.1. The flow chart of the process is illustrated in Fig. 4-6.

In the second stage, the changes of spring constant (kg/mm) were studied by blocking and pressing the spring from initial length to
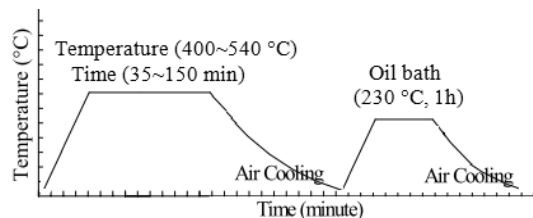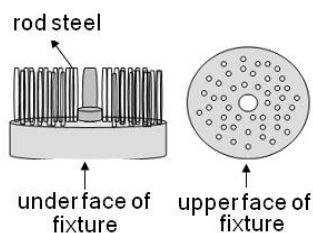


**Fig. 1.** Heat Treatment Cycle for Spring

**Fig. 2.** Schematic picture of block for spring steel 1,4568 (X7CrNiAL 17-7)



**Fig. 4.** Schematic path of dislocation and influence deposits in deformation of matrix and create obstacles against their movement [1]



**Fig. 3.** Apparatus measurement spring coefficient constant (force changes in terms of length)



**Fig. 5.** Diagram depending on changes of spring constant in terms of time at 400°C to 540°C temperatures

secondary length or block length (18 and 21 mm). The springs were placed into the fixture to block the springs (as sown Fig (2)) after after precipitation-hardening heat treatment (480°C for 45, 60, 70, 80 min), then were placed into oil bath at 230 °C for 1hr. The main reason of using this type of fixture is to prevent from deformation of the springs during blocking. The spring constant were measured with measurement hardness device in accordance to Fig(3). The spring constant is obtained by using this device by measuring the force changes ($\Delta F$) in terms of distance.

In the third stage, the spring constant changes in terms of length were evaluated at constant time and temperature without blocking. And finally the samples that were heat treated at 480°C for 80 min and were aged at the 230°C temperature for 1h in oil bath without blocking (this state is the optimum condition for spring production) were prepared for additional analysis.

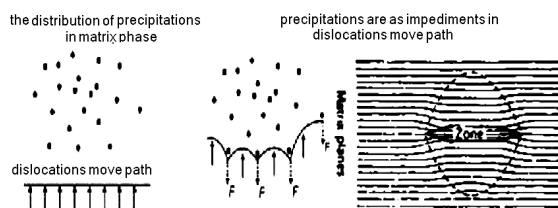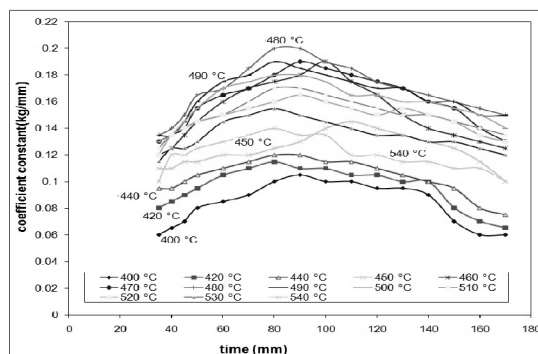The general microstructure of the material was observed by an optical microscope and SEM (Scanning Electron microscopy). Metallographic samples from selected heat treatment conditions were prepared and etched in chernical etching (the etchant consists of nitric acid, acetic acid and glycerin) to reveal the microstructures [4, 11]. The analysis of the base metal characteristic were obtained with the EDX and XRD.

## 3. RESULTS AND DISCUSSION

### 3. 1. Evaluation of Spring Constant Changes With Time and Temperature

As shown in Fig (5), the spring constant changes at 400 °C- 540 °C temperatures from 35 to 170 min shows a peak at each of temperature. With prolonging holding time at these temperature, the precipitation of inter-metallic compounds of Ni-Al ($Ni_3Al$ and $NiAl$) and carbides ($Cr_{23}C_6$) are increased, which were precipitaed in grain boundries from

supersaturated solid solution phase, so the hardness and strength of spring with the conventional precipitation strengthening rnechanism are increased[12]. The amount of precipitation depend on the time and temperature.

The Ni$_3$Al phase known as γ' phase, has ordered cubic, and phase NiAl has a cubic crystal lattice. Intermetallic γ' phase precipitate as coherent precipitate [13]. Coherence shows high stability of precipitates, that means very low surface energy per unit area of the precipitate/matrix. Precipitation hardening is achieved by the precipitation of coherent second phase from a supersaturated solution created by an aging heat treatment. The intermetallic phases formed impede the dislocation movement and therefore strengthening the alloy [1,15].

The mechanism of strengthening by precipitation hardening involves the formation of coherent clusters of solute atoms (that is, the solute atoms have collected into a cluster but still have the same crystal structure as the solvent phase). This causes high amount of strain because of mismatch in size between the solvent and solute atoms. The cluster stabilizes dislocations, because dislocations tend to reduce the strain. When dislocations are trapped by coherent solute clusters, the alloy is considerably strengthened and hardened. However, if the precipitates are semi-coherent, incoherent or are incapable of reducing strain behavior, a dislocation can circumvent the particles only by bowing into a roughly semi circular shape between them under the action of an applied shear stress. Consequently, the presence of the precipitate particles, provide higher strength by obstructing and retarding the movement of dislocations. The characteristic that determines whether a precipitate phase is coherent or non-coherent is the closeness of match or degree of disregistry between the atomic spacing's on the lattice of the matrix and on that of the precipitate[16].

Figure (5) shows that spring constant is reached to a maximum value by heating for 80 min at 480°C temperature and the maximum constant spring is low in below and above 480°C temperature.

In the temperature range between 400°C-480°C, the amount of Ni$_3$Al, NiAl and M$_{23}$C$_6$

precipitation in grain boundaries increases gradually, because the above mentioned precipitation particles are stable in this range[17,18,19].

With increasing temperature from 400°C to 480°C, the amount of precipitation particles at grain boundaries and matrix phases as barriers against of dislocations movement increases and thus the hardness and strength of springs increases.

With increasing temperature from 480°C to 540°C is facilitated the conditions for diffusion and aggregation of the precipitation particles in matrix phase and result in non-uniform distribution of the precipitation particles.

This non-uniform distribution of precipitation particles in matrix phase and also their aggregation in special places, is caused to removal obstacles against dislocations movement and therefore some dislocations are pile-upped in back of the obstacle, will release and continue to their movement. So the hardness and strength of spring reduced and therefore the spring constant decreased (Fig. 4). Therefore, a gradually increase of spring strength and constant with increasing temperature to 480°C, while a gradually decease of spring strength and constant with increasing temperature to 520°C is showed in Fig. 5.

According to Fig (5) the maximum spring constant for temperatures 460, 470 and 490 °C are seen in time 75, 90, 100 min, respectively. But the height of these peaks are lower than the 480°C temperature and 80 min.The time to acheive the maximum spring constant is decreased by increasing the temperature of heat treatment from 460 to 490°C.

The results show that the strength and spring constant distribution occures at range of time and temperature, but any peak is not seen in the temperature ranges lower than 460°C or higher than 490°C.

The changes of spring constant with primary length are shown in Figs (6) and (7). The spring are heated at 480°C temperature and are blocked to amount of 18-21mm and then heated at 230°C temperature for 1h in oil bath.Three major factors are determined the strength and spring constant are stress (applied force), temperature and
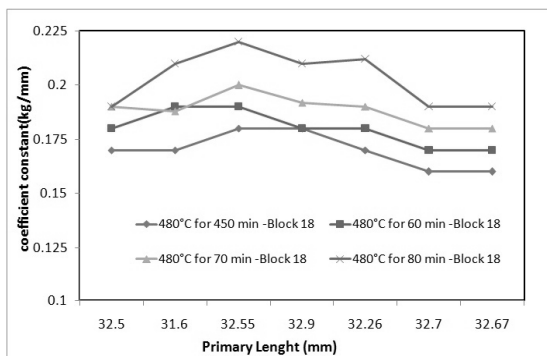
**Fig. 6.** Diagram depending on changes of spring constant in terms of primary length with 18mm block at 480°C and 45 to 80 min.
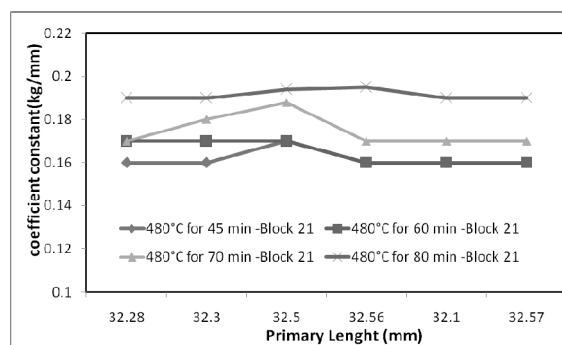


**Fig. 7.** Diagram depending on changes of spring constant in terms of primary length with 21mm block at 480°C and 45 to 80 min.

heating time.

Figures (6) and (7) shows that the spring constant changes with heat treatment at 480°C at times of 45 to 80 min for 18 and 21mm spring. As shown, the spring constant for 18 mm spring length after blocking more than 21mm, so, the amount of applied stress is the effective factor in strenghting of the spring. For austenitic stainless steels, as the cold work increases, the optimum aging temperature decreases[16].

With increasing heating time to 80 min at 480°C, the spring constant increases for both of blocking springs, but the amount of this effect is high in the 18mm length of spring after blocking. As expected, increasing the amount of cold work will cause the final spring constant to increase, as



**Fig. 8.** Diagram depending on changes of spring constant in terms of primary length with 18mm & 21mm block at 480°C and 45 to 80 min.

seen in Fig (8). For 17-7 PH, as the cold work increases, the optimum aging temperature decreases. According to Fig (8), the spring with 18mm block shows more spring constant at low temperatures and reached the optimum at 480°C temperature for 80 min. As the heating time is increased, the spring constant increase to a maximum, and then drop off.

The decrease in spring constant signifies a condition that is called "over-aging". Over-aging occures as the particles that caused the increase in strenght continue to grow in size. As these particle grow, they begin to coarsen and cause a decrease in the spring constant.

### 3. 2. Evaluation of the Spring Constant Changes With Time at Constant Temperature

The results of spring constant changes wit time at constant temperature without blocking are shown in Figs (9) and (10). In Fig (9) is observed that spring constant will gradually rise with increasing heat treatment time at 480°C temperature from 45 to 90 min and increasing heat treatment time from 90 to 150 min is decreased again. In Fig (10), at 520°C temperature, the spring constant was increased from 45 to 80 min and then was faced with severe downfall from 80 to 150 min. The diagrams of spring constant changes with time at 480°C and 520°C temperatures, indicate that the maximum spring constant is attainable about 80 min at 480°C temperature and is about 70 min at 520°C
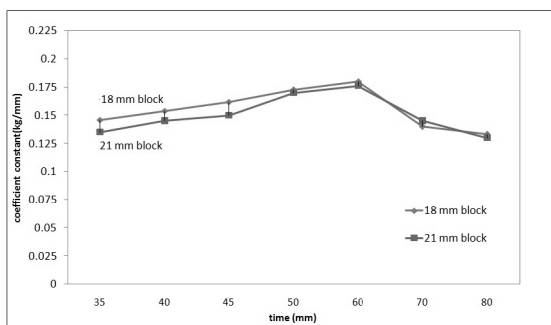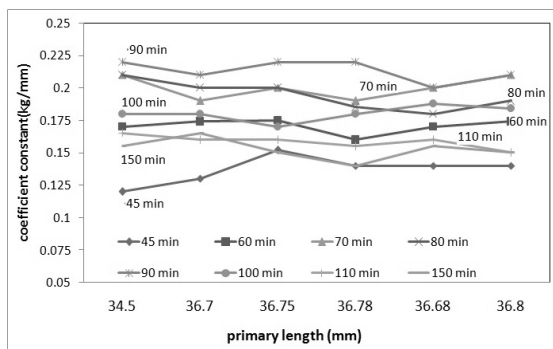
**24**

**Fig. 9.** Diagram depending on changes of spring constant in terms of primary length without blocking at 480°C and times (a) 70 (b) 80 min.
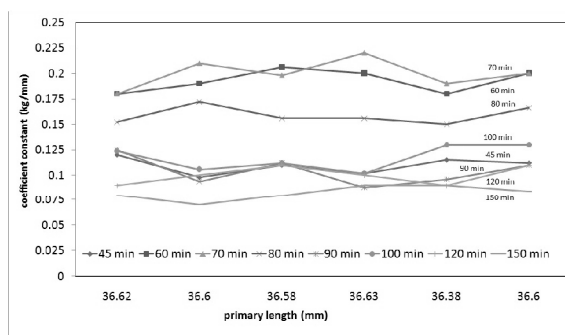


**Fig. 10.** Diagram depending on changes of spring constant in terms of primary length without blocking at 520°C and times (a) 70 (b) 80 min.
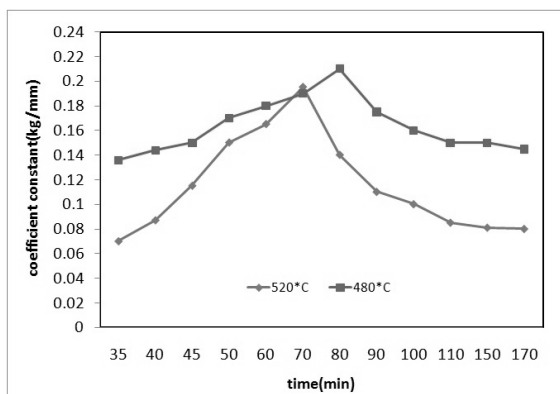


**Fig. 11.** Diagram depending on changes of spring constant in terms of heat treatment time without blocking (a) 480°C (b) 520°C, respectively.
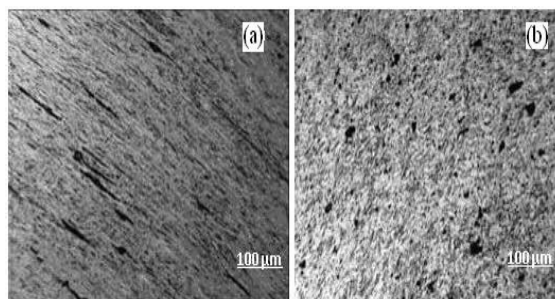


**Fig. 12.** Image of drawn austenite texture in directions (a) cross section (b) longitudinal section of spring steel 1.4568

temperature, respectively (Fig.11). With increasing precipitation hardening time and temperature the amount of precipitations in grain boundaries and also inside grains increases. With increasing heat treatment temperature, the required time to reaching maximum spring constant is decreased.

### 3. 3. Evaluation Microstructure PH17-7 Spring

The microstructure images were obtained on transverse and longitudinal direction after heat treatment of 17-7PH steel at 480°C for 80 min and aging at 230°C for 60 min in oil bath without blocking (Fig. 12). In these microstructures, the elongated grains in wire forming direction is observed. Due to the cold forming process on this

spring, the amount of internal stresses are high. Whatever, the elongated intermetallics were participated inside matrix phase as shown in the cross-section.

The analysis and characteristic of the base metal and components were obtained With EDX and XRD (Fig.13). SEM images of microstructure in Figs (14a,b) show typical compounds in matrix phase such as A and B Areas. The presence of metallic elements such as Ni, Al, Cr and Fe was confirmed using EDX and XRD in the A and B Areas (Figs. 15 and 16).

These Areas are created by precipitation of Ni-Al inter-metallics ($Ni_3Al$ and NiAl) and carbides such as $Cr_{23}C_6$ in matrix phase. The precipitated particle was elongated in forming direction in local Areas so the crystal lattice was more
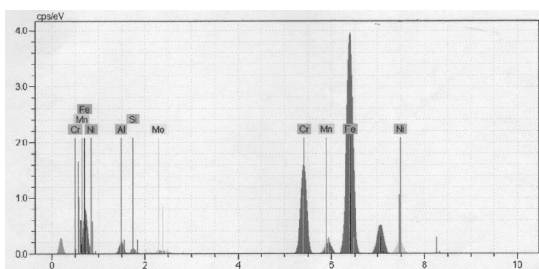
**25**

**Fig. 13.** EDX analysis of the spring steel 1.4568 microstructure after heat treatment (at 480°C for 80 min and cooling in air, then heating inside oil bath 230°C for 60 min and cooling to cold water).



**Fig. 14.** Formation of inter-metallic compounds in microstructure of matrix phase for spring steel 1.4568 after heat treatment (at 480°C , 80 min and cooling in air, then heating in oil bath 230°C for 60 min and cooling in cold water) and respectively (a) Scanning Electron Microscopy (SEM) (b) optical microscopy.



**Fig. 15.** EDX analysis combination A Area of spring steel 1.4568 microstructure after heat treatment (at 480°C , 80 min and cooling in air, then heating in oil bath 230°C for 60 min and cooling in cold water)



**Fig. 16.** EDX analysis composition B Area of spring steel 1.4568 microstructure after heat treatment (at 480°C , 80 min and cooling in air, then heating in oil bath 230°C for 60 min and cooling in cold water).

irregulared and these factors create a lot of big and large obstacles in front of dislocations movement.The results showed that with prolonging time of heat treatment at 480°C to 80 min, the number of these Areas with inter-metallic compounds and carbids ($Ni_3Al$, NiAl and $C_{23}C_6$) increases[20]. From 80 to 150 min, thess Areas and mentioned compounds decreases which adversly effects on strength and spring constant of samples.

## 4.CONCLUSIONS

1.    The results showed that heating temperature and time and stress factors (applied force) playing a major role in the spring constant.
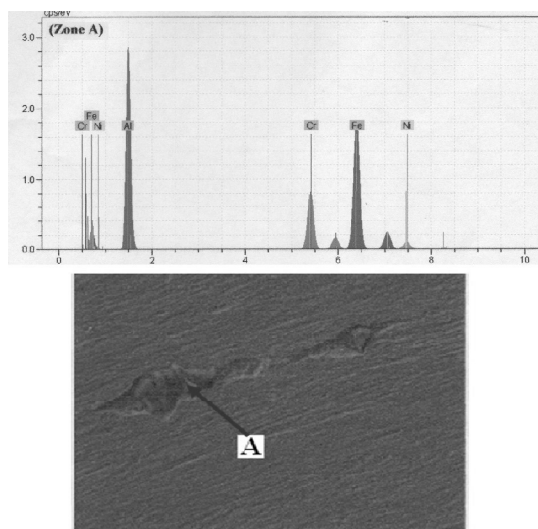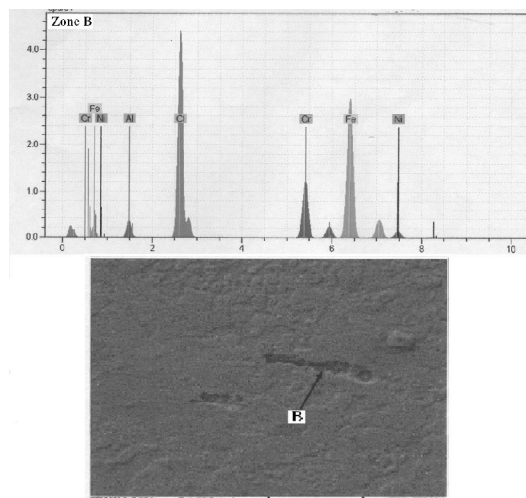
2.    The maximum of spring constant is obtained at 480°C temperature for 80 min for PH17-7 springs.

3.    The use of 18mm block shows more spring

constant than 21mm block.

4. The XRD and EDX analysis confirm the presence of metallic elements such as Ni, Al, Cr and Fe that these elements precipitate in matrix phase as $Ni_3Al$ and $NiAl$ and $Cr_{23}C_6$ inter-metallic combinations.

5. The results showed that with prolonging time of heat treatment at 480°C temperature to 80 min, the number of Areas with inter-metallic combinations increases and with prolonging time from 80 to 150 min these Areas decreases.

## REFERENCES

1. ASM HANDBOOK Vol.01, "Properties and Selection Irons", Steels, and High-Performance Alloys , Formerly tenth Edition, Library of Congress Cataloging in Publication Data American Society for Metals,1990.

2. The Atlas Specialty Metals Technical Handbook of Stainless Steels, 2003.

3. Wang, D., Kahn, H., Ernst, F. and Heuer A. H., "NiAl precipitation in delta ferrite grains of 17-7 precipitation-hardening stainless steel during low-temperature interstitial hardening". Scripta Material, 2015, 108, 136–140.

4. Faki, B. and ubela, D. "Influence of heat treatment of 17-7PH steel with modified chemical composition", 18th International Research/Expert Conference (TMT '2014), Budapest, Hungary, 2014.

5. Johnson, C. R. and Grimsley, J. D., "Heat treatment for improved stress-corrosion resistance of 17-7 PH stainless steel", NASA, TN D-4673,1968.

6. Rimac, M., Oruč, M. and Beganović, O., "contribution to the development of high-strength steel through reasearch of the relationship between microstructure and properties" 15th International Research/Expert Conference, Trends in the Development of Machinery and Associated Technology(TMT 2011), Prague, Czech Republic, 2011,12-18.

7. Craig, R. G., Slesnick, H. J. and Peyton, F. A., "Application of 17-7 Precipitation-hardenable Stainless Steel in Dentistry", J DENT RES,1965, 44, 587-595.

8. Ziewiec, A., Zielinska, A. and Tasak, E., "Microstructure of welded joints of X5CrNiCuNb16-4 (17-4 PH) martensitic stainles steel after heat treatment", Archives of metallurgy and materials, 2014, 59, 965–970.

9. Yip, T., "Cyclic Plasticity of 17-7 P-H Semi-Austenitic Stainless Steel, A thesis submitted in conforrnity with the requirements for the degree of Doctor of Philosophy", Graduate Department of Metailurgy and Materials Science University of Toronto,1998.

10. Peckner, D., Bernstein, I. M., "Handbook of Stainless Steel", Published by McGraw Hill, Inc., New York, NY, U.S.A., 1977, 7- 11.

11. Mills, K., ASM Handbook. Volume 9. Metallography and microstructures,, Formerly Ninth Edition, ASM International,1985.

12. BAS EN 10088-3:2007 - Stainless steels. Technical delivery conditions for semi-finished products, bars, rods, wire, sections and bright products of corrosion resisting steels for general purposes.

13. Faki, B., Buri, A. and Kratina, E., "Effect of aging on microstructure of 17-7PH steel with modified chemical composition", 19th International Research/Expert Conference ,Trends in the Development of Machinery and Associated Technology, (TMT 2015) Barcelona, Spain, 2015, 22-23.

14. Hüller, M., "Optimization of Composition and Heat Treatment of Age-Hardened Pt-Al-Cr-Ni Alloys", Metallurgical and materials transactions A, 2005,36,683-685.

15. McGuire, M. F., "Stainless Steels for Design Engineers", ASM International, 2008.

16. Murathan, Ö. F. and Abul, M. R., "Heat Treatment of 17-4 PH Stainless Steel", MATE 405, Term Project submitted to Prof. Dr. Erdoğan TEKİN in partial fulfillment of the requirements for MATE 405 Heat Treatment and Surface Hardening of Materials, 2011.

17. Smith, W. F., "Structure and Properties of Engineering Alloys", 1993.

18. Fakić, B. and Ćubela, D., "Review of the development of research in the design of semi austenitic stainless steel 17-7PH", 17th International Research/Expert Conference (TMT 2013), 2013, 113-116.

19. Fakić, B., Ćubela, D., "Modification of

chemical composition and thermal treatment of semiaustenitic stainless steel 17-7PH", Metalini I Nemetalni Materijali, Bugojno, 2014, 21-22.

20.  Chen, C. W., "The structural evolution during low temperature carburization of 17-7 PH stainless steel", Submitted in partial fulfillment of the requirements For the degree of Master of Science, 2012.