Material Property Characterization for Low Pressure Carburization Process Design using Computer Modeling

Zhichao (Charlie) Li, Justin Sims
and B. Lynn Ferguson
DANTE Solutions, Inc.
Cleveland, Ohio, USA

Jason Fetty and Treven Baker
US Army
Combat Capabilities Development Command
Aviation & Missile Center (CCDC AvMC)
Fort Eustis, VA, USA

ABSTRACT

Low Pressure Carburization (LPC) is widely used in the aerospace industry for hardening components made of steels with high alloy content and high heat resistant properties. The traditional gas carburizing process often generates Intergranular Oxidation (IGO) near the surface due to the existence of oxygen in the furnace atmosphere, which needs to be removed by grinding after hardening to restore bending and contact fatigue performance. LPC processing is done in a low pressure chamber without the existence of oxygen, so the surface microstructure is improved by eliminating IGO. High temperature resistant steels require high alloy element contents, and some elements are strong carbide formers, such as Cr, Mn, Mo, and V, etc. During LPC processing, both iron and alloy carbides can be formed, which significantly affect the carburization time required to reach a specified case depth and surface carbon. The carbides formed during the LPC process may not decompose completely prior to quench hardening, and these primary carbides will end up in the final processed parts. If the size of these primary carbides is not controlled, both bending and contact fatigue performance may be decreased. In order to control carbide formation during LPC, the carbon diffusivity of a material must be characterized. This characterization was recently performed under a program between DANTE Solutions and the Combat Capabilities Development Command Aviation and Missile Center (CCDC AvMC). In this research, a specifically designed coupon was used to characterize the carbon diffusivity and carbide forming properties during LPC processes. Using the characterized material properties, LPC process recipes can be designed by using modeling to achieve specific case depth and surface carbon content. The work was demonstrated using Pyrowear 675 steel and DANTE commercial heat treatment modeling software.

INTRODUCTION

Critical components in aerospace propulsion systems often favor using high temperature resistant steels. These steels are designed to have high tempering temperatures above 480° C (900° F). To achieve high temperature resistant properties, a high alloy content is used to form stable alloy carbides at high temperature. Pyrowear 675 (P675) is one of these high temperature resistant steels, and its nominal chemical composition is listed in Table 1.

Table 1. Nominal chemical composition of P675.

C	Mn	Si	Cr	Ni	Mo	V	Co	Fe
0.07	0.65	0.4	13.0	2.6	1.8	0.6	5.4	Bal.

P675 contains strong carbide forming elements, including Cr, Mn, Mo, and V, which makes the carburization process difficult. To reduce the amount of carbides formed during carburization, a furnace temperature of 900° C (1650° F) or lower is typically used for gas carburization of P675. Surface preoxidization is also used prior to gas carburization to reduce the alloy content on the surface, so the amount of carbide formation is controlled. Alloy carbides tend to form and grow with more available carbon, and the carbides tend to decompose if the carbon is depleted. The amount of nascent carbon on the part surface is controlled by the atmosphere carbon potential using a furnace oxygen probe during gas carburization, and the part surface carbon can be reduced by increasing the oxygen content of the atmosphere. The adjustment of the carbon potential is not instantaneous though, and carbon will flow out of the part surface due to chemical reactions with atmosphere oxygen when the carbon potential is low [Ref. 1]. In summary, the amount of nascent carbon in the part surface is difficult to control flexibly during a gas carburization process. Different from gas carburization, the LPC process uses a series of boost/diffuse steps, as shown schematically in Figure 1. During a boost step, the carbon is introduced into the part surface by flowing acetylene in the furnace chamber. During a diffuse step, the acetylene is pumped out, and the furnace chamber is in a vacuum

---

Presented at the Vertical Flight Society's 75th Annual Forum & Technology Display, Philadelphia, PA, USA, May 13-16, 2019.

condition, so there is no carbon flux in or out of the part surface. The carbon content on the surface decreases with the carbon diffusing inward from the surface, and the amount of surface carbon is controlled by the time of diffusion. The carbides formed during this process are more flexibly controlled than gas carburization by adjusting the time durations of the boost and diffuse steps. Because surface carbon and carbides can be more flexibly controlled, the furnace temperature used for an LPC process can be higher than that of a gas carburization process. Using P675 steel as an example, 900° C (1650° F) is often used for gas carburization, while 954.5° C (1750° F) is acceptable for an LPC process. As shown in Figure 1 schematically, the X-axis represents the processing time, and the Y-axis represents the partial pressure of the carbon gas of the carburization chamber. During a boost step, acetylene is introduced into the chamber, and acetylene cracks on the part surface to provide nascent carbon. A typical boost step may last between 30 seconds to several minutes. A carrier gas may be used to make the acetylene flow in the chamber more uniform. During a diffuse step, the acetylene is pumped out of the chamber. With the chamber being in a vacuum condition, there is no carbon flux in or out of the part surface. The carbon content on the part surface decreases during the diffuse step. Once the surface carbon drops below a certain value, the next boost step will start. In general, the time duration of the diffuse steps should increase as the LPC process proceeds, as shown in Figure 1.

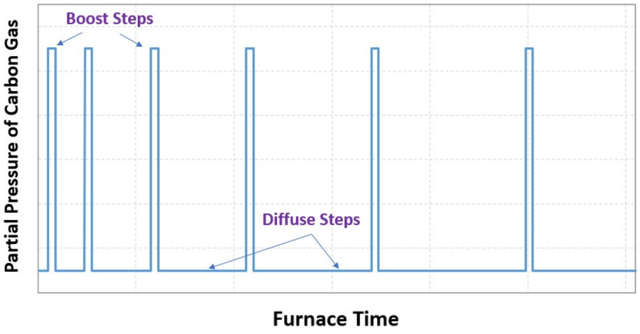
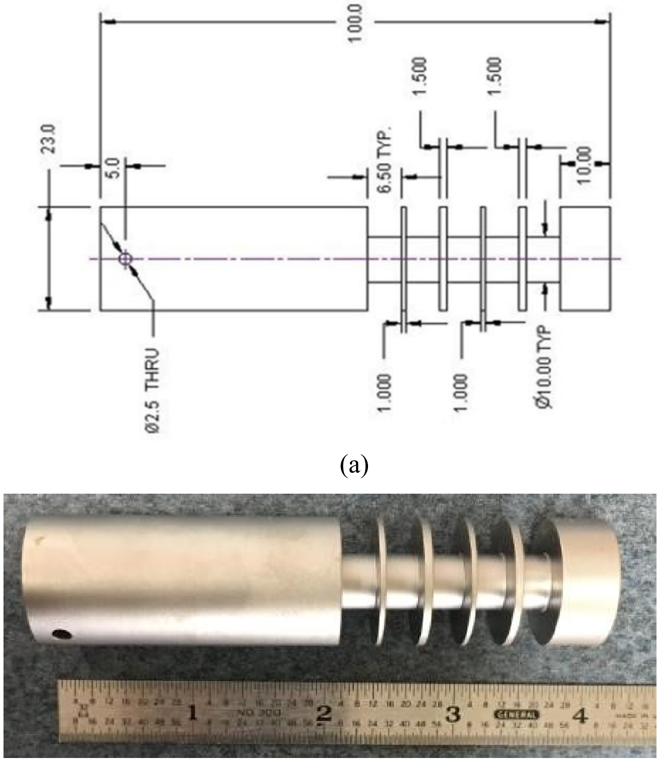


Figure 1. Schematic plot of Boost/Diffuse schedule for LPC process.

SAMPLE PREPARATION AND HEAT TREAT EXPERIMENTS

DANTE Solutions designed a coupon to characterize the material properties needed for modeling the LPC process. Factors considered for the custom coupon design included: 1) easy machining, 2) consistent results from LPC processing, 3) ease of carbon measurement, and 4) convenient for microstructure and microhardness characterization. The drawing of the designed LPC coupon is shown in Figure 2(a), and a picture of the machined coupon is shown in Figure 2(b). The outer diameter of LPC coupon is 23 mm, and the total length is 100 mm. Thin disks are used for measuring the amount of carbon which entered the disks and checking their hardness and microstructure. The disks have two sizes, 1.0 mm and 1.5 mm. The average carbon into the 1.5 mm disk was expected to be higher than that into the 1.0 mm disk from the same process, while the averaged carbon content was expected to be lower in the 1.5 mm disk due to its larger mass. Two disks and the end stud were designed for hardness and microstructure characterization. The end stud was designed to represent the hardness and microstructure found in the large cylinder end.

Using the measured carbon distributions of various LPC processed coupons, two categories of data were characterized: 1) amount of carbon and rates into the part surface during boost steps, and 2) the carbon diffusivity and carbide forming/decomposing properties in terms of different carbon values. The measured carbon distributions of the processed coupons from various LPC schedules were fit into the carbon diffusivity properties and carbide forming/decomposing model parameters in a format which could be used by the DANTE carburization models [Ref. 2-3]. With the validated model and material properties, LPC process recipes were designed to achieve specified effective case depth and surface carbon.



(a)

(b)

Figure 2. (a) LPC coupon drawing (unit: mm), and (b) Picture of fabricated coupon.

Twenty (20) LPC furnace runs were planned under this study, and two coupons were used for each run. Two carburization temperatures were used: 954.5° C (1750° F) and 1010° C (1850° F). For each furnace run, a unique LPC processing recipe was designed using DANTE computer modeling with the intermediate material properties fit from the obtained experimental data. The accuracy of the fitted material properties was improved upon as more experimental data was obtained.

After LPC processing, the coupons were quenched using 2 bar nitrogen, followed by a deep freeze at -100° C (~150° F), and tempering at 485° C (~900° F). Figure 3 shows two coupons in a tray for LPC and high pressure gas quench (HPGQ) processing. The coupons were hung vertically in the tray by a thin wire, which prevents the possibility of contact with the tray and blocking the carbon flux into the surface.

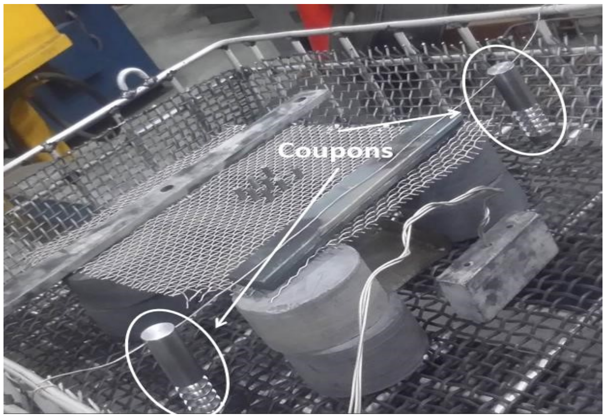


Figure 3. Two LPC coupons in a tray for LPC and HPGQ process.

LPC PROCESS MODELING AND BOOST/DIFFUSE RECIPE DESIGN

Computer modeling was used for both LPC recipe design and data fitting. A 2D axisymmetric model was developed using the cross-section shown in Figure 4(a) by assuming a uniform carburization boundary condition in the circumferential direction of the coupon. The finite element meshing is shown in Figure 4(b), and it contains 23,285 4-sided linear elements. The average mesh size is 0.25 mm, and finer elements are used in the surface layer of the coupon to catch the carbon gradient during the LPC process. A magnified view of the surface mesh is shown in Figure 4(c).

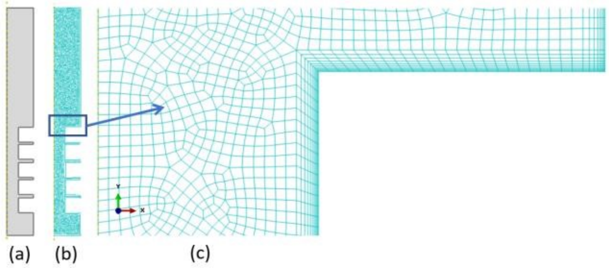
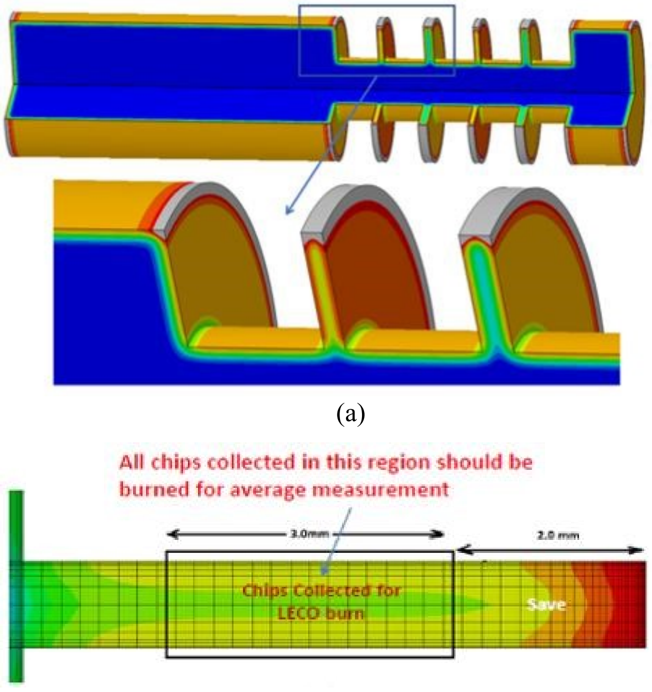


Figure 4. (a) 2D section of the LPC coupon, (b) Finite element meshing, and (c) Magnified view of the mesh showing fine elements used in near surface region.

Figure 5(a) shows the predicted carbon distribution contour from one LPC process model. The carbon content on the corner of the coupon is higher, as shown by the grey color in the contour. The reason for the higher carbon value on the corner is because the carbon flux into the corner is from two surfaces, which is considered a geometry effect. If the surface to mass ratio is higher, the surface will end up with higher carbon content. For example, a concave shape (gear root) has a lower carbon content than that of a convex shape (gear tooth flank) from the same LPC process. An example of the predicted carbon contour is shown in the lower Figure 5(a), and it clearly shows: 1) the thinner disk has higher carbon content than the thicker disk, and 2) the outer surface of the disks has higher carbon content. The carbon content of the processed coupons was measured using LECO burns. For the large cylinder, the carbon values were measured in term of depth from the OD surface. However, the mass of the disks allows only the averaged carbon content to be measured. To reduce the corner effect, 2 mm of material from the OD of the disks was discarded, and the chips collected for the LECO measurement are between 2 mm to 5 mm depth, as shown in Figure 5(b). The carbon distribution in the thickness direction of the disk is not uniform, and the predicted carbon is averaged to compare with the experimental data.


(a)

(b)

Figure 5. (a) Example of predicted carbon distribution contour of LPC coupon, and (b) Region of thin disk used to collect chip to measure carbon content.

As described in Figure 1, the LPC process contains a series of boost/diffuse steps to control the amount of carbon into the part surface, and the amount of carbon diffusing away from the surface inward. If the boost time is too long, carbides will grow and form large, stable carbides on the surface, which is detrimental to fatigue performance. If the diffuse time is too long, the surface carbon can be too low to achieve the required hardness, which may also increase the furnace time unnecessarily. By using computer modeling, the carbon value and carbide amount in the part are predicted during the entire LPC process. Figure 6 shows an example of predicted carbon and carbide evolution at a near surface point of the coupon during boost/diffuse steps. During a boost step for P675 steel, carbides are formed in the surface. The carbides decompose and provide a carbon source for the diffuse step. It is important to accurately design the boost/diffuse step durations to provide enough carbon and to decompose carbides effectively.

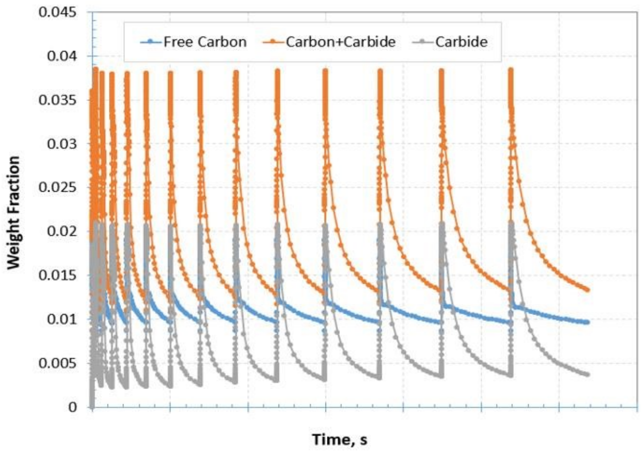


Figure 6. Predicted surface carbon during an LPC run showing carbon that is in solution and carbon that is tied up as a carbide.

RESULT AND ANALYSIS

Th planned twenty (20) LPC furnace runs and data collection have been completed. Using the measured data, the carbon diffusivity and carbide forming/decomposing properties were fit into DANTE carburization models. Two LPC processing temperatures, 954.5° C (1750° F) and 1010° C (1850° F), were used to generate material properties and boundary conditions for the DANTE carburization model. As shown in Figure 7, the measured carbon distributions are compared with the model predictions. Two coupons were processed for each of the furnace runs at Solar Atmospheres. The measured carbon distributions in terms of depth are from the long cylinder OD surface. Experiment-1 and Experiment-2 are the carbon distribution data measured from the two coupons processed together at 954.5° C. Experiment-3 and Experiment-4 are from two coupons processed at 1010° C. Prediction-1 is the modeling result for Experiment-1 and Experiment-2, and Prediction-2 is the modeling result for Experiment-3 and Experiment-4. The average carbon measured from the thick disk from the 954.5° C process is 0.71%, compared to the predicted value of 0.75%. For the thin disk from the same process, the measured and predicted carbon values are 1.03% and 1.02%, respectfully. The experiments and the predictions agree well. All twenty furnace runs used different recipes, either with different boost/diffuse schedules, or at different temperatures. Figure 7 only shows the comparison between experiments and predictions for two of the twenty furnace runs. However, all twenty furnace runs were compared between the experiments and predictions, and they all agree reasonably well. The horizontal dash line in Figure 7 represents a constant carbon value of 0.23%, which will give a hardness value of about 50 HRC with the current hardening and tempering conditions. A hardness of 50 HRC is considered the effective case depth (ECD) in gear hardening processes.

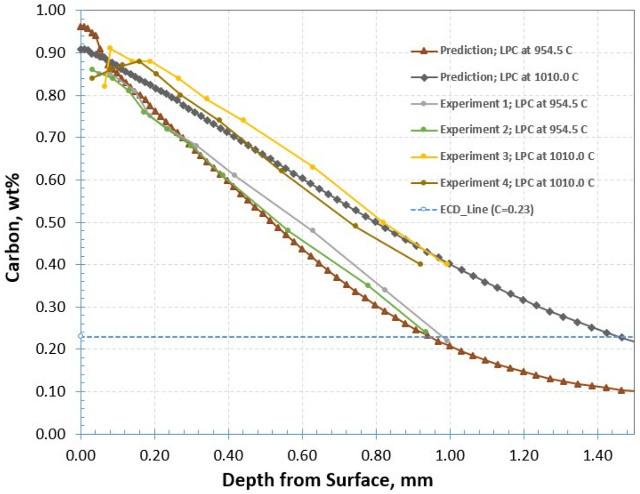


Figure 7. Comparison of predicted and measured carbon distribution from two LPC processes at 954.5° C and 1010° C.

In Figure 7, the same boost/diffuse schedule was used for the two LPC furnace runs at 954.5° C and 1010° C processing temperatures. The ECD from the 954.5° C process is about 0.95 mm, and the ECD from the 1010° C process is about 1.45 mm, which is about 50% deeper. P675 is known for low carbon diffusivity rates, and longer LPC processing time is required when compared to most other alloy steels. It is important to reduce the furnace time as much as possible, while maintaining acceptable microstructure and hardness.

To improve bending and contact fatigue performance, the carbon content and hardness of the carburized surface should be designed for specific applications. In general, HRC 60 or above is required on the surface for good contact fatigue performance. Slightly lower hardness on the surface may benefit the bending fatigue performance. Experiments in this study have been successfully used to correlate the relation between hardness values and carbon content for P675 steel based on the current austenitizing and tempering schedules. For martensite tempered at 485° C, 0.23% carbon has a hardness of ~HRC 50, and 1.1% carbon has a hardness of ~HRC 60. The two LPC experiments shown in Figure 7 at 954.5° C and 1010° C were designed to have a surface carbon of about 0.9%. Figure 8 shows a representative microstructure at a point near the OD surface from the Experiment-1 coupon. For both processes, the amount of carbides in the case is low, and the hardness values are below HRC 60. To achieve hardness values above HRC 60, higher surface carbon content is required. A higher austenitizing temperature may also be used to increase the hardness by reducing the amount of primary carbides. However, this was not covered in this study.

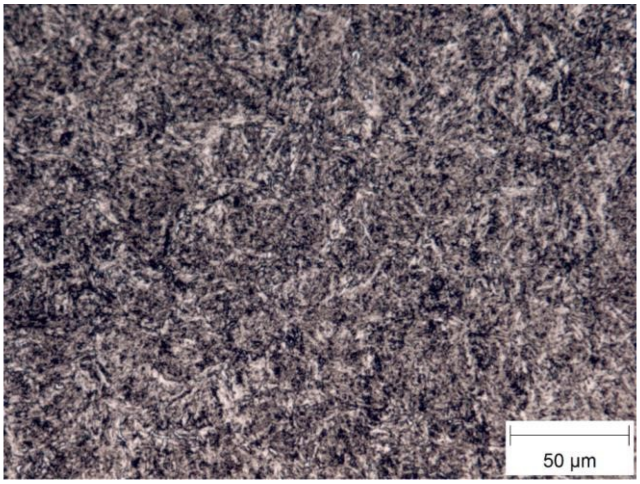
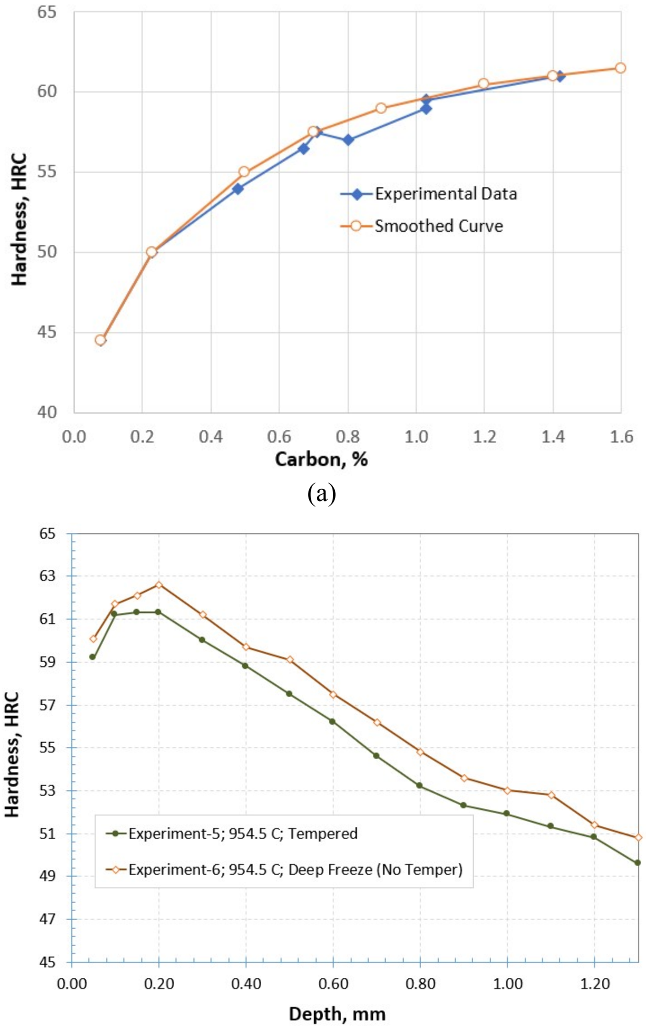


Figure 8. Microstructure at near OD surface from Experiment-1 process at 954.5° C (1750° F).

With the obtained carbon and hardness data from all furnace runs, the relation between the hardness of tempered martensite and the carbon content is obtained, as shown in Figure 9(a). The blue curve with diamond marks is the averaged hardness data from multiple experiments, and the orange curve with hollow marks is the fitted data. The curves shown in Figure 9(a) suggest that a carbon content of 1.1% or higher is required to achieve a hardness of HRC 60. Primary carbides are defined as those carbides formed during carburization, and they are not dissolved during reheating for hardening. It is expected that the reheating temperature and time will affect the amount of carbides dissolved and the hardness of tempered martensite in the final part. Figure 9(a) is limited to the current process used in this study, with a reheating temperature of 1037.8° C and a tempering temperature of 485° C.

By modifying the LPC boost/diffuse schedule, hardness values over HRC 60 were achieved on the surface with higher carbon content, as shown in Figure 9(b). The LPC process temperature shown in Figure (b) was 954.5° C (1750° F). During the heat treatment process, an extra coupon was used to check the hardness change caused by tempering. The extra coupon went through the exact LPC, HPGQ, and deep freeze processes as the other coupons, but it was pulled out before tempering for microstructure and microhardness checking. The comparison of the hardness values before and after tempering showed that the tempering practice used in this study will decrease the hardness by approximately 1.5 HRC points. By comparing the microstructures between tempered and as-quenched coupons, it was found that tempering is effective in reducing the carbide network microstructure along the grain boundaries. The hardness distribution shown in Figure 9(b) shows a lower hardness right on the part surface, and peaks at a depth of about 0.2 mm. The exact reason for this is unknown, but the possible reasons are described briefly: 1) effect of the measurement point being too close to the surface, 2) possibility of tensile residual stresses in the shallow surface, and 3) high amount of primary carbides in the shallow surface have no or minimum contribution to hardness.

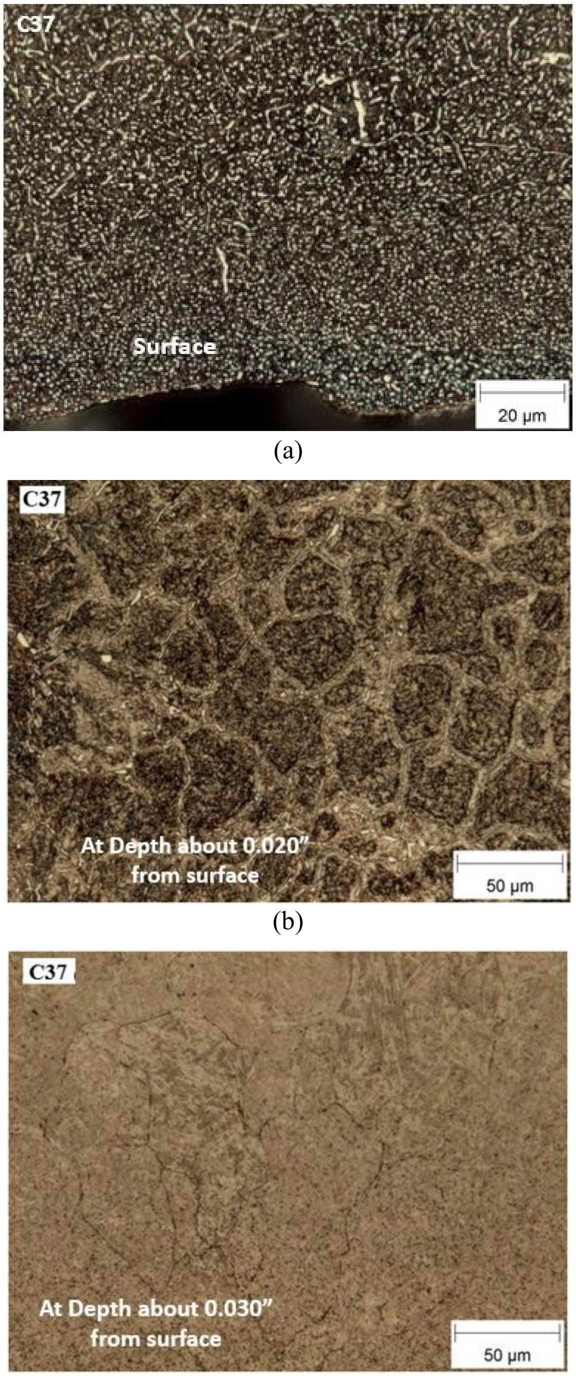


(a)

(b)

Figure 9. (a) Relation between hardness and carbon content obtained from 954.5° C (1750° F) LPC process, and (b) Example of hardness distribution obtained after LPC and HPGQ hardening process.

The microstructures of the tempered martensite from the Experiment-5 process at 954.5° C are shown in Figure 10, with Figure 10(a) representing the surface microstructure, Figure 10(b) representing the microstructure at ~0.5 mm (0.020”) depth, and Figure 10(c) representing the microstructure at ~0.75 mm (0.030”) depth. The surface has a high amount of carbides, and it is believed that they are mostly primary carbides. This conclusion was reached by comparing the microstructure differences between the tempered and as-quenched microstructure, noting the carbides looked like carbides in the as-quenched coupon. The surface microstructure shown in Figure 10(a) is considered as a typical carburized microstructure of P675 steel.



(a)

(b)

(c)

Figure 10. Microstructures from Experiment-5 process at 954.5° C (1750° F): (a) at near surface, (b) at depth about 0.5 mm (0.020"), and (c) at depth about 0.75 mm (0.030").

Figure 10(b) is the microstructure at about 0.5 mm depth from the OD surface, which is also a typical microstructure of P675 with medium carbon. The grain boundary has more alloy carbides than inside the grains. The carbides on the grain boundary are in broken form, which is an acceptable microstructure for P675 steel. At a depth of ~0.75 mm (0.030"), there is no grain boundary carbides found, as shown in Figure 10(c). Fine dispersed carbides are found inside the grains, and they are formed during tempering.

Figure 11 shows the microstructure, at a depth from the OD surface of about 0.5 mm, from the coupon processed at 1010° C (Experiment-7) using the same LPC boost/diffuse schedule as Experiment-5. By comparing Figure 10(b) and Figure 11, using 954.5° C and 1010° C LPC processing temperatures, respectfully, it is concluded that the carbides are in broken form for both cases. There is also no significant difference in the grain size between the two cases. Further evaluation of the microstructures at the near surface showed a higher amount of primary carbides for the 1010° C processing temperature, with no noticeable microstructural difference at any deeper depth. One conclusion is that 1010° C should be an acceptable LPC processing temperature for P675, but the boost/diffuse schedule should be adjusted from the 954.5° C boost/diffuse schedule because of the higher carbon diffusivity at 1010° C and because of the increased carbide formation during the boost steps.

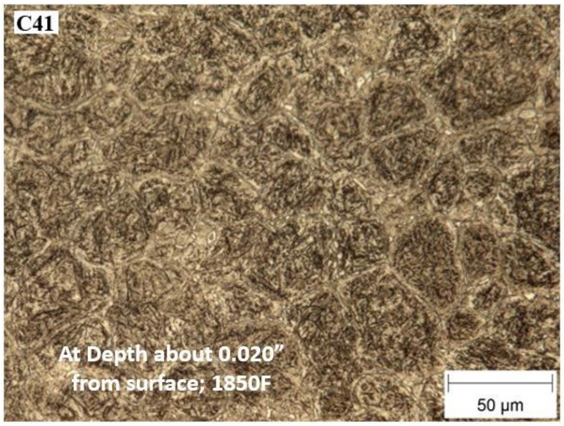


Figure 11. Microstructure at a depth of 0.020" from Experiment-7 process at 1010° C (1850° F).

APPLICATION OF LPC PROCESS DESIGN AND CARBON DISTRIBUTION USING MODELING FOR GIVEN PARTS

One goal of this study was to design LPC boost/diffuse recipes to meet the heat treatment specification and predict the carbon distribution for given parts. With the characterized material properties and carburization boundary conditions obtained from this study, the following specifications should be considered as LPC process design goals for any given part geometry using computer modeling:

- Surface carbon and surface hardness.
- Effective case depth (ECD).
- Variation of carbon distribution on different locations of the given part.

Figure 12 shows a CAD model of a bearing case made of P675 used to demonstrate the LPC process design and modeling. The outer diameter of the bearing case is 200 mm, the inner diameter is 170 mm, the height is 30 mm, and a fillet radius of 1.0 mm is used for all the corners. During the LPC process, the entire surface is carburized.

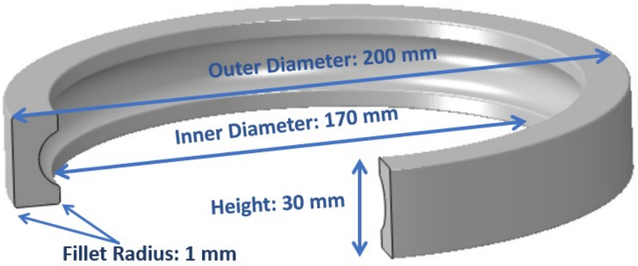


Figure 12. CAD model and brief dimensions of a ball bearing case used for demonstrating LPC process design carbon distribution modeling.

The heat treatment specification requires a hardness of HRC 59 or above on the ball path surface, with an ECD about 1.0 mm. In this demonstration, the selected carburization temperature was 954.5° C, and the carburization boundary conditions characterized from Solar Atmospheres' production furnace were used. It should be noted that different furnace types will behave differently due to the type of gas used, the effective time required for the carbon gas atmosphere to reach equilibrium, and the effective time to vacuum the chamber at the beginning of diffuse steps, and other factors. Therefore, a furnace should be characterized before it can be used to design the LPC process using computer modeling.

Using computer modeling, the designed LPC process recipe contained six (6) boost/diffuse steps, and the total furnace time was 12 hours. The predicted carbon distribution contour is shown in Figure 13.

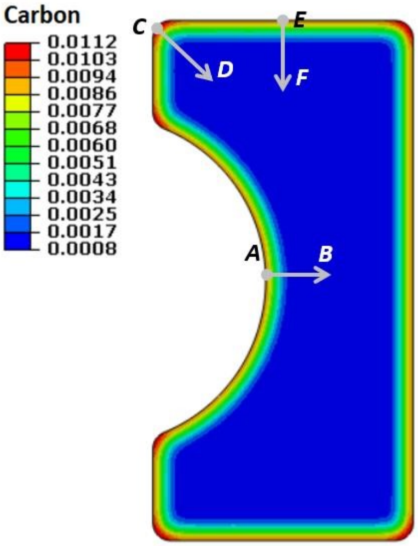


Figure 13. Predicted carbon distribution contour using the designed LPC recipe at 954.5° C.

A fillet radius of 1.0 mm was used for all the corners, and higher carbon values were predicted on the corners. The size of the fillet radius affects the severity of corner effect, and the fillet dimension can be adjusted in the allowable range to minimize the corner effect. As shown in Figure 13, three (3) locations were selected to post-process the carbon distributions in terms of depth from the surface. The three locations are labeled as, "AB" representing the ball path surface, "CD" representing the corner with 1.0 mm fillet radius, and "EF" representing the top flat surface.

The results of carbon distribution in terms of depth along the three lines "AB", "CD" and "EF" shown in Figure 13 are plotted in Figure 14 for the three locations. In this demonstration, the heat treatment requirements specify the minimum hardness of HRC 59 on the ball path surface, and the ECD is approximately 1.0 mm. To reach a hardness of HRC 59 after tempering, it is expected that the carbon content should be 0.9% or above, using the hardness and carbon relation in Figure 9(a).

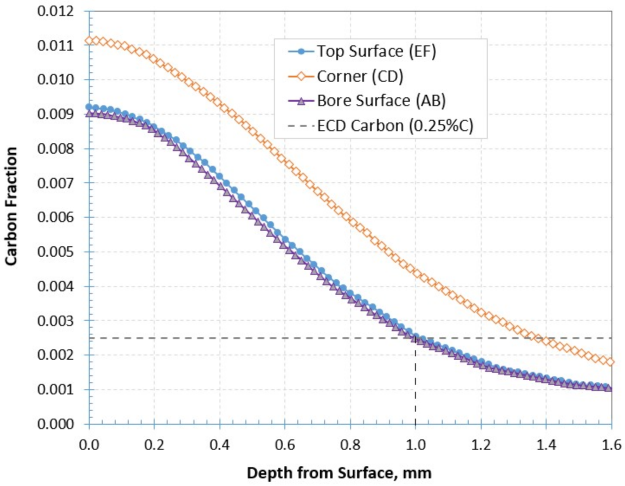


Figure 14. Carbon distributions in terms of depth from difference bearing case surfaces.

The purple curve with triangle marks in Figure 14 represents the carbon distribution along line "AB" from the ball path surface, and the predicted surface carbon is 0.90%, which meets the requirement. The predicted ECD of the ball path surface is about 1.0 mm using 0.25% ECD carbon criterion, which also meets the requirement. The carbon distribution along line "EF" from the top flat surface is plotted by the blue curve with round marks, and the carbon values are slightly above those of the ball path surface, which is caused by the geometry effect. All the corners have higher carbon values, and the carbon distribution along the line "CD" is plotted by the brown curve with diamond marks. The predicted surface carbon on the corner is 1.11%, vs. 0.90% on the ball path surface. The most effective way of reducing the high carbon phenomenon on the corner surface is by using a larger corner fillet or chamfer. Carburization of selective surfaces only using copper plating to block the noncarburized surfaces is often used to reduce the corner effect in aerospace industry.

This study also found that the carburization temperature and LPC boost/diffuse schedule will affect the severity of the high carbon phenomenon on the corner.

CONCLUSIONS

In this study, a series of LPC furnace runs were designed and executed to characterize the carbon diffusivity and carbide forming/decomposing properties of P675 steel at two processing temperatures, 954.5° C and 1010° C. Using the characterized data, computer modeling can be used to design the LPC process recipe and predict the carbon distribution including the geometry effect for given parts. The study has found that different furnace types produce different carbon distribution results from the same recipe. Therefore, the furnace behavior should be characterized before designing boost/diffuse recipes using computer modeling. The carbon diffusion rate is much higher at 1010° C than that at 954.5° C, and the achieved ECD is about 50% higher for 1010° C processing. The experiments didn't find noticeable microstructure difference between the results from the two carburization temperatures, including both grain size and surface carbides. It is suggested from this study that 1010° C LPC carburization temperature for P675 steel is acceptable. However, the boost/diffuse recipe at 1010° C should be adjusted from 954.5° C process due to its higher carbon diffusivity and higher amount of carbides formed during boost steps. Using the characterized material properties of P675 steel and carburization boundary conditions of Solar Atmosphere's production furnace, the LPC process design and carburization process prediction were successfully demonstrated using DANTE software. DANTE can therefore be used to successfully design the low pressure carburization process for components made out of P675.

Author contact: Zhichao (Charlie) Li, Charlie.Li@Dante-Solutions.com

ACKNOWLEDGMENTS

The views and conclusions contained in this document are those of the authors and should not be interpreted as representing the official policies, either expressed or implied of the U.S. Government. Authors also wish to acknowledge Solar Atmospheres, Inc. for heat treating the experimental coupons, and Tensile Testing Metallurgical Laboratory for measuring the carbon profiles, checking microstructure and hardness of processed coupons.

REFERENCES

1. Herring, Daniel H., and Robert V. Peters Jr., Technical and Process Advantages of Low Pressure Vacuum Carburizing Using Chemical Acetylene with DMF Solvent, Gear Technology, September 2013

2. Geoffrey Parrish, "Carburizing: Microstructures and properties", Year 1999, ISBN: 978-0-87170-666-9

3. Lynn Ferguson, Zhichao Li. Justin Sims, and Tianyu Yu, "Vacuum Carburization Steel Alloys Containing Strong Carbide Formers", Year 2017, Proceedings of the 29th ASM Heat Treating Society Conference, At Columbus, Ohio, USA