Plasma Carburizing of Tungsten†

Kazuhiro NAKATA*, Kazuhiko KAWAMURA**, Tomoya KITADA***, Katsumi KANBE**** and Masao USHIO*****

Abstract

A new DC glow discharge plasma carburizing process by RF induction heating enabled the high temperature carburizing of tungsten at 1773K, and a preferred formation of W_2C to WC as carbide was confirmed at higher temperature than 1573K with an atmosphere of Ar+40% CH_4 mixture gas at 133Pa.

KEY WORDS: (Plasma carburizing)(RF induction heating)(Tungsten)(Carbide)(Tungsten carbide) (Structure)(Amorphous carbon)(Growth kinetics)

1. Introduction

Carbides of refractory metals have attractive properties such as high hardness, high melting point, low work function, good heat and electric conductivities, so on. Due to embrittlement of carbide, however, it is difficult to make a bulky material except for WC-Co super alloy. If the surface of the refractory metal can be changed to carbide, however, the industrial application of these materials will be extended. Carburizing is an important surface treatment for tungsten electrodes to modify the electrode property, since W_2C carbide is effective for extending the electrode life time [1]. In tool industries, tungsten rods with surfaces converted to carbide, if possible, have high potential the applications as thorough-hole punches.

A DC glow discharge plasma carburizing process has been applied to steel materials to achieve good controllability of the structure of the carburized layer. However, the application of plasma carburizing to refractory metals is rare, because high temperature treatment is difficult and the highest treatment temperature available in an industrial furnace is around 1373 K.

N.Y.Pehlivanturk and O.T.Inal [2] and N.Kanayama et al [3] reported the carburizing of tungsten by a DC plasma carburizing process without and with carbon heater assistance, respectively at the carburizing temperature between 1173 K and 1373 K. They reported a thin carbide layer formation and that a WC layer was dominantly formed in the carbide layer, and the W_2C layer was negligibly thin.

The purpose of this study was to develop a new plasma carburizing apparatus in which a RF-induction heating system is employed to heat a specimen to high temperature during DC glow discharge, and to create a thick W_2C layer on the surface of the tungsten.

2. Experimental Procedures

Figure 1 shows a schematic illustration of a plasma carburizing apparatus, which is a conventional DC glow discharge plasma nitriding/carburizing apparatus, but additionally equipped with a RF-induction heater of 100 kHz and 10 kW. An industrially pure sintered tungsten rod, 7 mm in diameter and 60 mm in length was set on a cathode specimen holder. DC glow discharge was applied to it with a constant discharge power of 0.3 A and 300 V in an atmosphere of Ar+40vol%CH_4 at 133 Pa. The specimen was mainly heated by induction heating and specimen temperature was measured by a non-contact infrared thermometer. Carburizing temperature and time were varied from 1373 K to 1773 K for up to 7.2 ks. After carburizing treatment, the specimen was cooled by Ar flow in the chamber. Mean cooling rate from carburizing temperature to 773 k was very fast, around 600 K/min.

† Received on December 4, 1998

* Associate Professor
** Chubu Electric Power Co.,Inc.
*** Nihon Densi Kogyo Co,Inc.
**** Graduate Student
***** Professor

Transactions of JWRI is published by Joining and Welding Research Institute of Osaka University, Ibaraki, Osaka 567-0047, Japan.

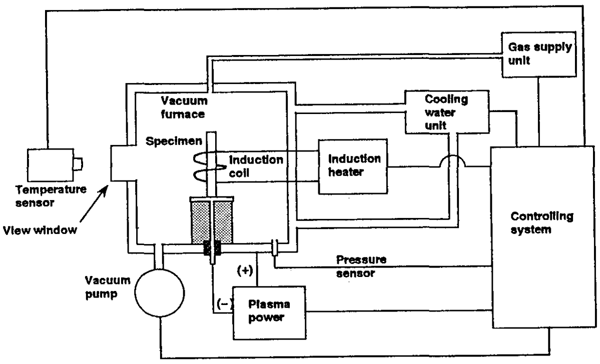

Fig.1 Schematic illustration of DC glow discharge plasma carburizing apparatus equipped with RF-induction heater.

Microstructure of the carburized specimen was revealed by electrolytic etching with 0.5%NaOH water solution and observed by SEM, and analyzed by EPMA. Analyses by X-ray diffraction(Cu-Ka) and Raman spectrometer were used to identify the structure.

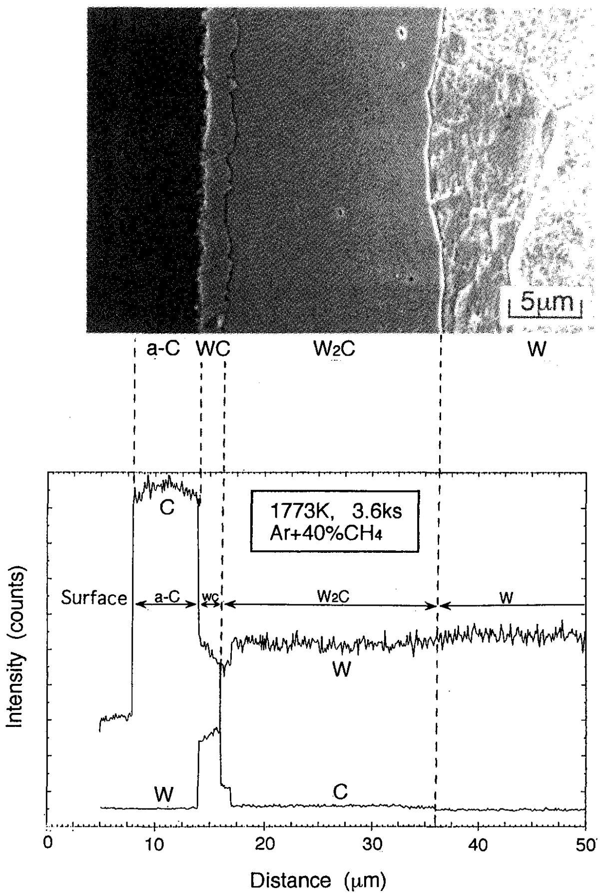

Fig.2 SEM micrograph and EPMA result showing C and W element distributions on cross-section of carburized specimen.

3. Results and Discussions

3.1 Microstructure

Figure 2 shows a typical SEM micrograph on a cross-section of a carburized specimen at 1773 K and the result for line analysis of carbon and tungsten (W) . From the surface, three layers appeared. The outer layer was black and consisted of carbon only. Raman spectrometry revealed it to be an amorphous carbon layer (a-C), which deposited on the specimen surface and was easy to peel off. The intermediate layer, 2-3 μm in thickness and the inner thick layer consisted of both carbon and tungsten, but the intermediate layer showed a much higher carbon level than the inner layer. The substrate tungsten as a specimen core was deeply etched and carbon distribution was not observed clearly.

Figure 3 shows the results of X-ray diffraction analysis on an as-carburized surface (a) and a partly eliminated surface of the specimen (b) as shown in a sketch (c). In (a), strong WC and weak W_2C peaks were detected as carbide phases together with very weak W peaks. W and W_2C peaks, however, became stronger than WC after the surface elimination. This means that W_2C

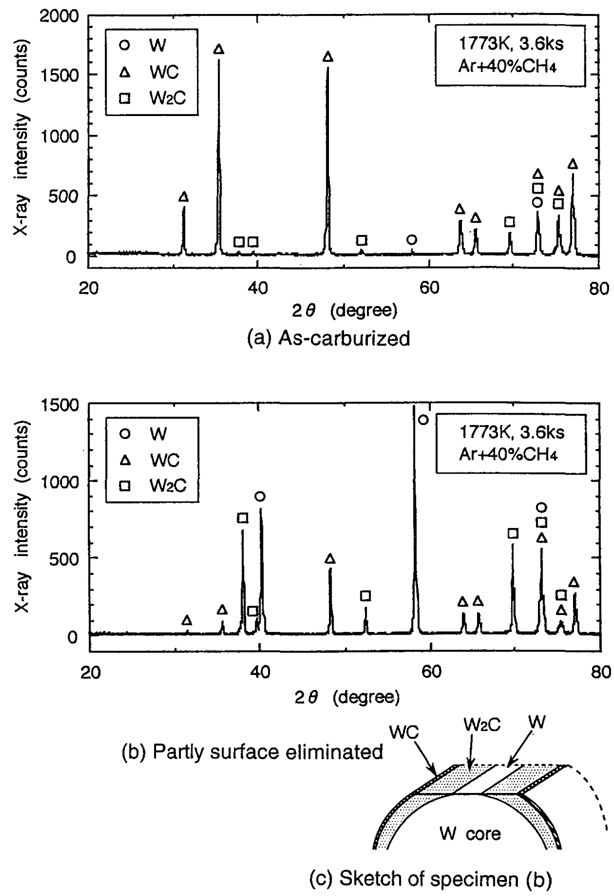

Fig.3 X-ray diffraction analyses on (a) as-carburized and (b) partly surface eliminated specimens.

layer was formed beneath the WC layer. Therefore, the intermediate thin layer and inner thick in Figure 2 were identified as WC and W_2C, respectively.

RF-induction heating without a glow discharge did not form any carbide and amorphous carbon layer.

3.2 Carburizing temperature

Figure 4 shows SEM micrographs of carburized zones at different carburizing temperatures. At 1673 K two carbide layers are clearly observed and the inner layer, W_2C became thicker than the outer layer, WC, and this was most clearly observed at 1773 K. On the contrary, at 1473 K an almost single layer of WC was formed and it was difficult to detect a very thin W_2C layer beneath the WC layer.

Figure 5 shows the relation between the thickness of each carbide layer and carburizing temperature for the carburizing time of 7.2 ks. The increase in WC thickness with carburizing temperature was very small, and WC thickness was thin, about 3 μm, even at 1773 K, which was only about two times that at 1373 K. On the contrary, an abrupt increase in W_2C thickness appeared at more than 1573 K and reached about 20 μm at 1773 K.

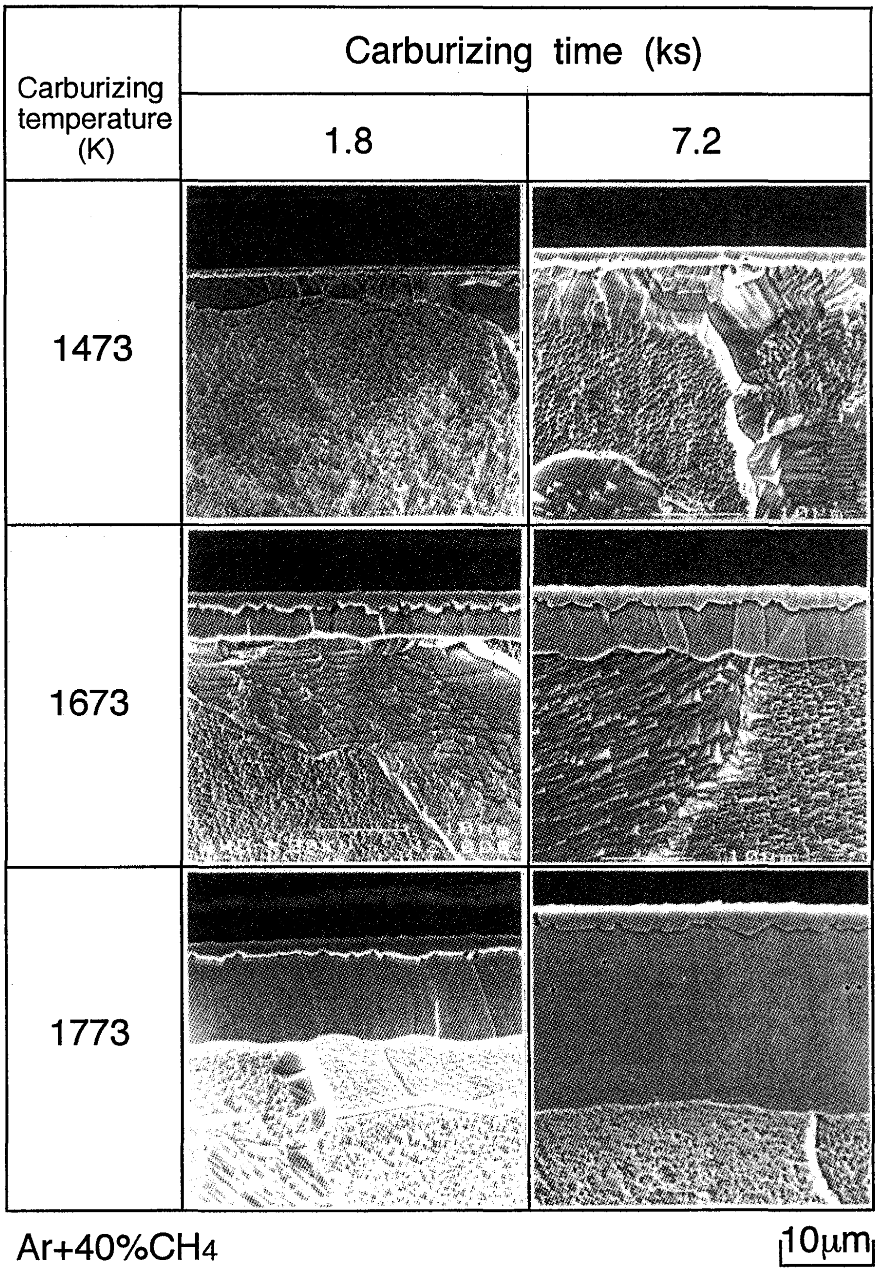

Fig.4 SEM micrographs on cross-sections of carburized specimens at different temperatures.

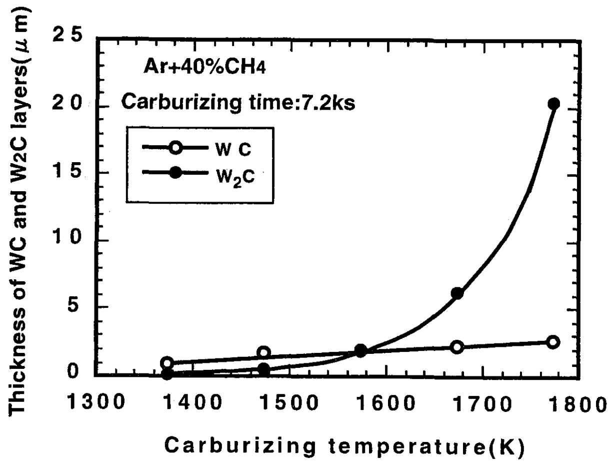

Fig.5 Effect of carburizing temperature on thickness of WC and W_2C layers.

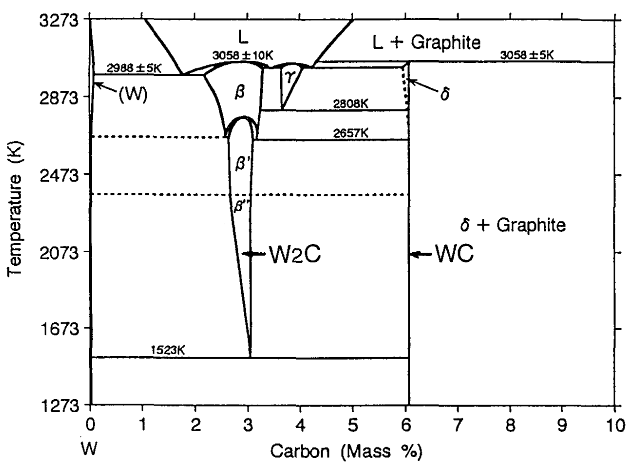

Fig.6 Equilibrium W-C binary alloy phase diagram.

According to the tungsten-carbon equilibrium phase diagram as shown in **Figure 6**[4], W_2C appears at temperatures higher than 1523 K, and below this temperature, it is unstable and WC is the only stable carbide phase.

Therefore, these results show a good agreement with the phase diagram. Due to the high cooling rate after carburizing, W_2C phase formed at high temperature remained down to room temperature, even though it was unstable at lower than 1523 K.

Results in the literature [2,3] showing a dominant formation of WC as a carbide at a carburizing temperature between 1173 K and 1373 K coincide with these conclusions drawn from the phase diagram.

Consequently, in order to make a thick W_2C layer, the carburizing temperature should be raised higher than 1523 K.

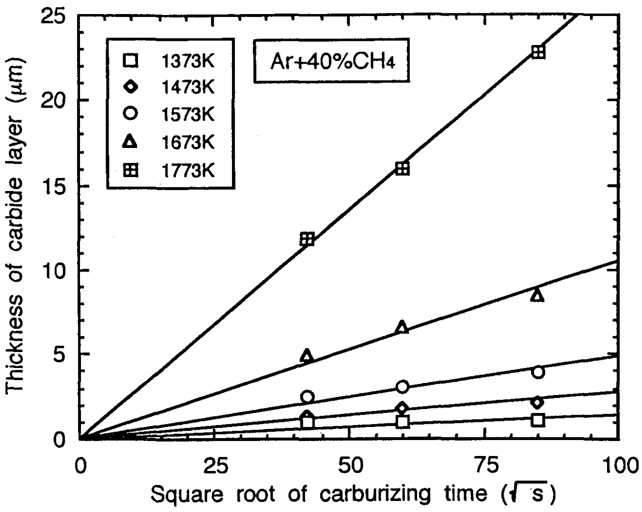

Fig.7 Relation between square root of carburizing time and full thickness of carbide layer ($WC+W_2C$).

3.3 Growth rate

Figure 7 shows the relation between the square root of carburizing time and the full thickness of the carbide layer ($WC+W_2C$) at different carburizing temperatures. Typical parabolic growths were observed at each carburizing temperature. These relationships indicate that the growth of carbide layer is controlled by volume diffusion.

4. Summary

DC glow discharge plasma carburizing of tungsten at temperatures as high as 1773 K became possible by assisted RF-induction heating during glow discharge. Two carbide layers, WC and W_2C formed, and a preferred formation of W_2C appeared at carburizing temperatures higher than 1573 K with an atmosphere of Ar+40%CH_4 mixture gas at 133 Pa.

Acknowledgment

Funding for this research was supported partly by Grant-in-Aid for Scientific Research (B)(2) No.08455338 from The Ministry of Education, Science, Sports and Culture.

References

1) S.Fukagawa: Vacuum Tube Materials and Ultra High Vacuum Engineering, Ohmusya, (1956), 118 (in Japanese).
2) N.Y.Pehlivanturk and O.T.Inal: Int. Conf. on Ion Nitriding, edi. by T.Spalvin, ASM International, Cleveland, OH, USA, (1986), 179.
3) N.Kanayama, Y.Horie and Y.Nakayama: ISIJ International, Vol.33 (1993), 615.
4) T.B.Massalski: Binary Alloy Phase Diagram, 2nd ed., ASM, (1990), 896.