Technical Report
Received January 11, 2002
Accepted for Publication January 25, 2002

Effect of Immediate Quenching after As-sintered on Microstructure of WC Phase and Strength of WC-14mass%TiC-8mass%Co

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WC-14%TiC-8%Co cemented carbide of as-sintered was immediately quenched. In comparison with conventional sintered specimen, the strengths of the quenched cemented carbides were increased by 20.2%~32.8%. It was ascribed that the formation of the slip steps on WC grain's edge is induced by thermal-stresses during quenching.

Key Words : carbides ; X-ray diffraction ; dislocation; mechanical properties ; transmission electron microscopy

Introduction

WC-TiC-Co cemented carbides are widely used as basic materials for cutting tools. Brittle refractory carbides of transition metal WC and TiC are combined with a tough binder metal Cobalt by powder metallurgical sintering method. Due to cobalt binder phase having the nature of isomeric transformation and variable solid solution line, the researches on composition, phase constitute, precipitation and its distribution in cobalt binder phase had been concentrated with regard to effect of heat treatment on cemented carbides [1-4]. Since hardness (HRA) stands for strength of alloys skeleton and WC grains are basically unvaried before and after heat-treatment, the investigations of WC hard phase were only concentrated in a residual-stress and a contiguity of WC grain. In general, WC phase occupies in above 70-mass percentage in whole cemented carbides, however it is not clear how heat-treatment influences the microstructure of WC and strength of whole alloy. In this paper, we employed WC-14mass%TiC-8mass%Co to investigate the effect of immediate quench on grain morphology, defects, microstructure of WC hard phase and the cemented carbides mechanical properties.

Table 1 Main characters of powders

Powder	C_{Total}	C_{free}	Fe	O_2	Ti	Fsss	Density
	mass%	mass%	mass%	mass%	mass%	μm	g/cm^3
WC	6.08-6.78	0.20	0.10	0.25	----	4.20	----
Co	<0.10	----	0.30	0.50	----	----	<0.75
(Ti,W)C	11.0-11.4	0.30	0.20	----	31.5-32.5	----	----

Table 2 Norminal composition, process parameters, physical and mechanical properties of alloys

	Norminal comp.			Process Parameters*		Physical and mechanical properties				
Spec.	WC	TiC	Co	Quench	Medium	Density	Hardness	Coercive force	TRS**	State
No.	mass%	mass%	mass%	T/K		g/cm^3	HRA	kA/m	MPa	
TS	78	14	8	----	----	11.6	92.3	11.9	1326.8	Sintered
Q1	78	14	8	1523	Oil	11.5	92.4	13.9	1594.7	Quenched
Q2	78	14	8	1573	Oil	11.4	92.4	14.2	1612.5	Quenched
Q3	78	14	8	1623	Oil	11.4	92.6	14.4	1762.4	Quenched
Q4	78	14	8	1673	Oil	11.2	92.5	13.4	1738.3	Quenched
Q5	78	14	8	1723	Oil	11.4	92.5	13.9	1754.0	Quenched

* : Sintering temperatures are all 1723K;
** : Transverse rupture strength, the average of 10 test pieces

Materials, experiment and procedure

The characteristics of commercial powders using for preparation of specimens are shown in Table 1. Processing from ball-milling powders to pressing bulk specimens has been prepared according to standard of ZYQ/A2002-86. Sintering and immediate-quenching the specimens have been carried out in WZX-20 type of vacuum furnace at 5x10^{-4}Torr. The composition and heat-treated parameters of cemented carbides are given in Table 2. Specimens of 5x5x35 mm were used for measuring transverse rupture strength (TRS) with three-point bending test. A slice specimen of 4x4x0.12 mm had been cut by wire-discharged machine at 3 kV. The spark-cut discs were mechanically polished to 70~80μm, then foils were etched by ion-milling the slices in LBS-1 type of ion mill for examination in the TEM, at 5x10^{-4}Torr, 4 kV and 10 μA of Argon ion beam. Microstructure of cemented carbides has been observed by using Hitachi 800 TEM equipped with EDAX 9100 and JEM-2010 at 200 kV. WC and (Ti, W) C powders were used as a stress-free reference standard. These powders were the same as that used to make the specimens. Microstress in WC and

(Ti, W) C was determined by Voigt function of a single peak X-ray diffraction method. WC (001) and (Ti, W)C (002) peaks were chosen for analyzing their microstress, with Siemens D-500 X-ray Diffractor at 36 kV and 30 mA using CuK α.

Experimental results and discussion

1. Effect of quenching temperature on mechanical properties

According to conventional testing methods, physical and mechanical properties of specimens are given in Table 2. Compared hardness, coercive force and TRS of quenched specimens with sintered specimen (TS), TRS of specimens firstly increase and then slowly decrease with increase of quenching temperature. Peak of TRS appears at 1623K, increasing rate at peak quenching temperature is 32.8%. The transverse rupture strengths of the cemented carbides are increased by 20.2%-32.8% in as immediate quenched than in as sintered. Rockwell Hardness of specimens fluctuates without deteriorating. Coercive forces of specimens slightly increase with increase of quenching temperature. Because changes in magnetic property of specimens are not relation with the size of test piece, it is that heat-treatment influences the whole volume specimens, heat-treatment to cemented carbides is a kind of whole volume strengthening method [4]. For required whole volume-strengthening WC-TiC-Co alloy used for intermittent cutting tools, the surface modification processing such as CVD and PVD coating cannot be substituted for heat-treating to the cemented carbides.

2. Effect of immediate-quenching on WC phase microstress

Owing to the difference in coefficient of thermal expansion (CTE) and elastic modulus between hard phase and binder phase, significant residual thermal microstress appears in various phases after cooling. Microstresses of WC and (Ti, W)C are given in Table 3. From the table, total microstresses of WC and (Ti, W)C in quenching specimens are higher than that of sintered. It is well known that quenching may results in deformity of crystal lattice and miniaturization of grain-block. The CTE of Co is about two times that of WC (i.e. $C_{Co} = 12.5\times10^{-6}$, $C_{WC} = 6.4\times10^{-6}$). And it is accompanied with the solid phase transformation of β-Co $\rightarrow \varepsilon$-Co during cooling, they induce microstress to form in various phases, and their microstresses rise up with increase of quenching temperature.

Table 3 XRD analytic results of microstress in WC phase

Spec. No.	Microstress, MPa WC	Microstress, MPa (Ti,W)C	Crystal block sige, μm WC	Crystal block sige, μm (Ti,W)C
TS	326.4	120.5	0.471	0.763
Q1	384.3	132.6	0.442	0.735
Q2	378.1	129.7	0.457	0.736
Q3	380.4	114.8	0.449	0.741
Q4	367.2	126.1	0.467	0.750
Q5	370.5	130.0	0.455	0.714

※: Calculated by E_{wc} = 722GPa, $E_{(Ti,W)C}$ = 322GPa & γ = 0.2

3. Effect of immediate-quenching on WC microstructure

Since WC mass percentage is over 70 in WC-TiC-Co alloys, WC grains come into contact with themselves, as shown in Fig. 1. It was observed that the sharp angles of WC polygonal grains in quenched specimens have become round and the edges of some WC grains have become round wave-like shape in Fig. 2(b). Especially, the edges of WC grains adjacent to Co binder phases appear some peculiar steps as indicated by arrow in Fig. 2(c). Rounding angles of WC grains with quenching after as-sintered very possibly results from WC dissolved in Co binder. The formation of wave-like edges of WC grains relates to the orientation of primary WC grain and quenching stress field. Rounding angles and forming wave-like edges of WC grains is an advantageous factor of improving strength due to decreasing stress concentration at a top angle of WC grains and increasing contact-area between WC and Co binder. Some peculiar steps of WC grains with quenching have been analyzed by using the trace-line method of TEM. As shown in Fig. 2, stretching direction of the step is almost parallel to intersection-line between diffraction crystal plane (010) and $(001)_{wc}$, that is trace line of [210]. It is well known that crystal plane {001} of hexagonal WC ($c/a = 0.976 \fallingdotseq 1$) is close-paced plane, when a WC grain was subjected to some kind of stress, mobile dislocations in WC grain can be accumulated along {001} plane as indicated by arrow in Fig. 3(a), the dislocations in WC grains strongly slip according to {001}<110> easier slip system (so-called {0001}<11$\bar{2}$0> slip system), it induced the slip step to formed. As shown in Fig. (d), peak identification with EDAX

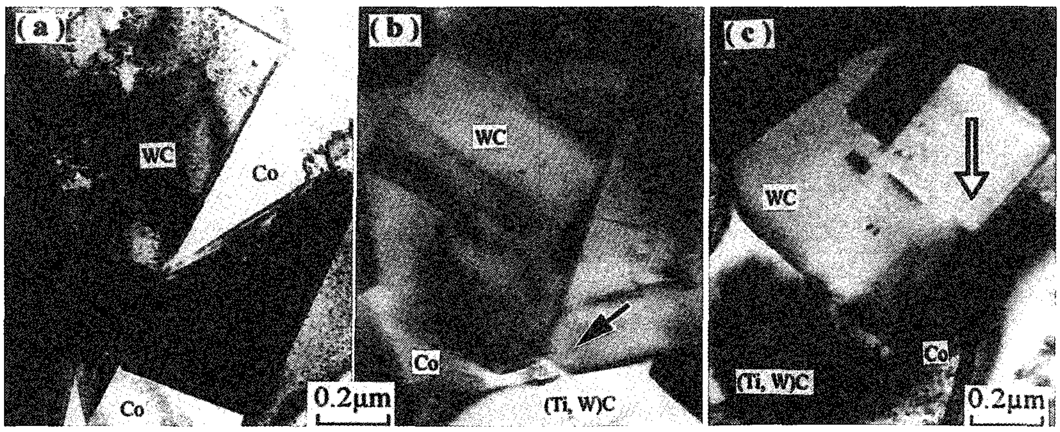

Figure 1 TEM diffraction-contrast images (BF) of WC phases in WC-14masst%TiC-8mass%Co. (a) WC grains showing the regular polygon in sintered specimen (TS), (b) showing the round-angle and wave-like edges of WC phases in quenched specimen (Q2), (c) showing a step on WC grains in specimen (Q4) as indicated by arrow.

9100 to this grain further verifies which the grain with slip is WC.

The kind of slip step's formation reason may be explained by a thermal stress mechanism. Due to existing large difference of CTE between WC and Co, WC phase is subjected to tensile stress and conversely Co binder to compressive stress [4]. During high temperature stage (approximately less than 1613K) of cobalt solidified, the WC grains were exerted by complex stresses, one among them may be described as Fig. 3. Thermal stresses above-mentioned easily enable WC grain to occur plastic deformation. During quenching of alloys there is a critical temperature (Tc) of plastic flowing [5], at the temperature, as cooling rate is higher and absolute value of stress in various phases is largest, it is also largest that shear stresses exert on WC grains. It results in that mobile dislocations (b_1=<0001>, b_2=<11$\bar{2}$0>/3 & b_3=<11$\bar{2}$3>/3) [6] in WC phase were moved and accumulated along {0001} close-packed plane in WC grain. Once the critical stress of slip required is reached, {0001}<11$\bar{2}$0> easier slip systems start, average amplitudes of slip enable to be up to 60 nm, which formed slip steps.

As a result, the steps enable decreasing possibility of nucleus formation of microcracks inside WC grain and increasing possibility of microcracks propagating along WC/Co interface. It is generally considered that main advantageous effect of heat treatment on the cemented carbide is improving phase constitute of Co binder, that is remaining more than fcc-Co contents with better coordinating deformation ability. But in practical using conditions of loading or raising temperature as cutting friction, the phase transformation from fcc-Co to hcp-Co can be take place, it will results in deterioration of Co binder action of coordinating deformation. However, WC slip steps do not disappear as loading or raising temperature of cutting, that is, its positive action to the cemented carbide enables maintaining. For WC-TiC-Co obtaining low cobalt, suitable heat-treatment to the cemented carbide not only improves the composition and phase constitute of Co binder phase, but also improves microstructure of WC hard phase, in the meanwhile both enable to increment of the cemented carbide's mechanical properties.

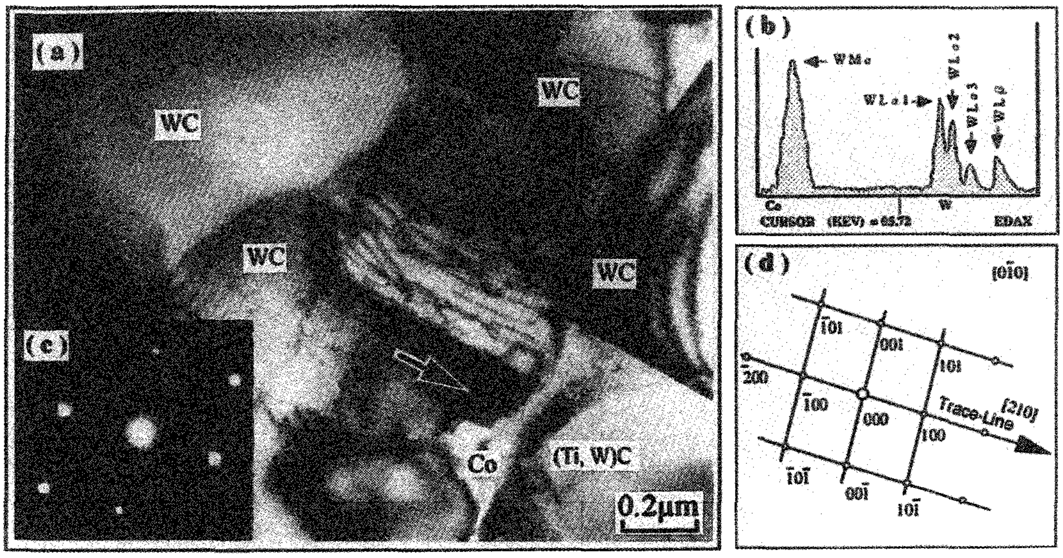

Figure 2 A step structure of WC grains in WC-14mass%TiC-8mass%Co (Q3) through immediate quenching. (a) the diffraction-contrast image, (b) EDS of WC phase with "steps", (c) diffraction pattern and (d) its indexing. The analyses indicating that "step" structure of this WC grain is a "Slip Step" formed according to <11$\bar{2}$0>{0001}.

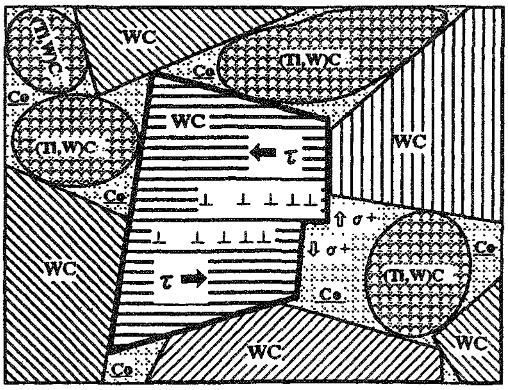

Figure 3 Schematic diagram of formation of slip step. In powder sintered specimens, i.e. specimen Q3, WC grains are subjected to complex stress actions as shown in above figure. Under elevated temperature of quenching and shear stress τ actions, the glissile dislocations have been made to accumulated along {0001} close-packed face of WC grains. Eventually, that <11$\bar{2}$0>{0001} slip system starts form the slip step.

Conclusions

The results obtained in the present work can be summarized as follows : For the first time, slip steps on WC grains adjacent to Co binder have been observed and confirmed by TEM in WC-14mass%TiC-8mass%Co cemented carbide of as-immediate quenched. Quenching stresses enable mobile dislocations to move and accumulate along {0001} close-packed planes in hexagonal WC ; thus strong slips start according to {0001}<11$\bar{2}$0> easier slip systems. As a result, the steps occur on WC grains adjacent to

Co binder, and the procedure is very possibly taken place in high temperature stage of as immediate quenching. Without deteriorating other mechanical properties, the transverse rupture strengths of cemented carbides increase by 20.2%-32.8% in as immediate quenched than in as sintered.

Acknowledgments

The authors are grateful to Senior Engineer J. B. Liu and Engineer J. Z. Guo, Zhuzhou Cemented Carbide Research Institute, P. R. China, for their helpful supports on the preparation of specimens.

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