Phenomenological characteristics of Microindentation in WC-Co- TiC+TaC Sintered Carbides

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Abstract: Hardness is the most important property in specifying sintered carbides. Considerably less work has been carried out in micro hardness range on sintered carbides. The main objective of the present investigation is to study the phenomenological characteristics of microindentation behavior in commercial sintered carbides, in particular, the microhardness Vs indentation load, rate of loading & unloading, indentation time, neighboring indentations, heating and cooling in atmosphere to 973K. A study of statistical distribution of micro hardness values is also carried out to investigate the possibility of developing micro hardness measurements in the load range of 0.1-1N as a quality control method. Certain interesting observations are made on the fringes that appear in the indentation and their removal with time after unloading indicating certain flow of material around the indentation for which plausible explanations are given.

Key words: Microindentation, Sintered carbides, Hardness, High temperature, loading and unloading

I. INTRODUCTION

It is well known that, the extensive use of sintered carbide alloys is characterized by high hardness. The wide use of cemented carbides arises because these materials have a unique combination of mechanical, physical and chemical properties, in addition to high hardness. The metallurgical characteristics of the alloys are such that, these properties can be substantially modified by composition and control of structure, so that they could be tailored into various grades for widely different conditions of service. One problem concerning the properties and uses of sintered carbides stems from the difficulty of translating values of the more easily measured properties into specific characteristics. This difficulty has thus led to the philosophy adopted by major producers of determining the alloy for a particular application by a process of experience and trail, while taking care that subsequent batches are held within close limits. This enables easily measured properties, such as hardness, to be controlled when used in conjunction with a microstructural assessment of quality.

Most of the investigations on sintered carbides are carried out in the macro hardness range of 10–1500N, which cannot indicate the structural features. Considerably less work has been carried out in the microhardness range. The present work has been carried out to investigate the possibility of developing microhardness measurements in the range of 0.1-1N as a QC method on sintered carbides. Since, microhardness is sensitive to micro structural features, such as shape, size and distribution of particles and plastic flow in a localized region, attempts were made to characterize the material interms of microhardness. Since the microhardness measures the resistance to indentation, it is subjected to statistical variations, elastic and plastic deformation in a localized region. Some of the interesting observations made regarding these factors have thrown further light on the mechanical behavior of sintered carbides in general. Thus micro hardness measurements have led to a greater understanding of the structural features and plastic flow of sintered carbides.

Sintered carbides are tough when compared to other materials with similar hardness of HV30. However, plastic deformation before fracture is considerably small, and there is a considerable scatter in the obtained values of tensile stress. A variation in the strength of about 1.5:1 in a sample of 25 specimens from the same batch is not uncommon. The distribution of results obtained is not symmetrical about mean but slightly long-tailed on the low strength side. The high strength side appears to tend towards a limiting value. The mean and standard deviation, therefore, provide a satisfactory comparison between like materials, provided, the asymmetry of distribution is not forgotten [10]. Almost every application of sintered carbides involves the generation of considerable amount of heat. It is therefore, clearly essential for the sintered carbides to possess good elevated temperature properties, which vary widely with both composition and grain size. A useful measure of thermal shock resistance is the ratio of thermal conductivity to thermal expansion. Sintered carbides have a particularly favorable combination of these properties when compared to other hard materials. This explains the superiority of sintered carbides based on the tungsten carbide, cobalt system over ceramics and cermets [10]. Sintered carbides have been widely used in the major group of applications such as, metal removal, metal forming, and mining/earth handling where, wearing of parts is a common phenomenon. However, in order to expand the use of sintered carbides, particularly in the field of structural components, a fundamental knowledge of the factors influencing the various properties would be required [9, 10].

Ivan'ko investigated the microhardness of carbides of some of the transition metals and suggested that, keeping the specimen under load for a long time may lead to reduction in the micro hardness values owing to vibrations, the use of very small loads may cause the results to be too high, while too high a load may give rise to brittle fracture of the material. Based on linear fracture mechanics, *Viswanandhan and Venables* observed a quantitative relationship between the palmqvist crack resistance parameter 'W', and bulk hardness

'H' of cermets, that can be expressed as, 1/W=(AH–B), where A and B are constants. These are meaningful comparisons between mechanical properties of various cermets [7]. Investigations in the fields of thermal fatigue, crack propagation and stress-strain behavior on W-Co composites are being conducted in several labs [11-13].

II. EXPERIMENTAL

2.1. Material

The composition (%wt.) of sintered carbide specimens used in the present investigation is given in Table.1. The specimens were polished to mirror like surface using diamond paste (0-2μm) and mounted in Bakelite to enable easy handling.

Table.1. Composition (%Wt) of sintered carbides specimens used in the present investigation

Specimen identification	Composition(Approx.)			Remarks made by
	WC	TiC+TaC	Co	
S1	75	15	10	Supplier 1
S2	75	15	10	Supplier 2
S2(a)	75	15	10	"
S3	Not known -		-	"

2.2. Load Vs microhardness

The Microhardness of test samples was measured using a microhardness tester consisting of Vickers pyramid diamond indenter. The hardness number is calculated using eq.1 given below.

$$H = 1.82\times10^4 (P/d^2) \quad \text{MPa} \qquad (1)$$

Where 'P' is applied load in Newton and'd' is average length of the two diagonals of the impression in microns. Microhardness measurements were done on S2&S3 at different loads ranging from 0.1-1N in steps of 0.1N. Each reading was an average of five readings. All the necessary precautions were taken during microharness measurements. The obtained microhardness values were plotted against applied load as shown in Fig.1.

2.3. Effect of rates of loading and unloading on microhardness

The microhardness of S2 was taken at different loads ranging from 0.2-1N in steps of 0.2N at constant indentation or contact time. At each load, the microhardness values were measured with different rates of loading and unloading. The rate of loading and unloading varied from 0.455- $4.55\times10^{-2}N.s^{-1}$. The variation in hardness as a function of rate of loading/ unloading at each load is plotted as shown in Fig.2.

2.4. Indentation creep

The microhardness of S2&S3 was measured with 1N load at different indentation times varying from 5 to 1000sec keeping the rate of loading and unloading constant. The obtained length of the diagonals was plotted against the indentation time as shown in Fig.3.

2.5. Statistical distribution of microhardness

The microhardness with 1N load was carried out, keeping the rate of loading/unloading and indentation time constant, in order to compare the microhardness distribution in S1&S2 with same composition, intended for same application supplied by different manufacturers. Around 100 readings were taken on each specimen and frequency curves were drawn for comparison of hardness variations in the two specimens as shown in Fig.4&5. A small area of $4mm^2$ was chosen on the specimen to study the hardness distribution.

2.6. Effect of heating on microhardness

The microhardness with 1N load was carried out on S2(a), keeping rate of loading/unloading, indentation time constant, in order to study the effect of heating in atmosphere to 973K for 1hr and further cooling to the ambient temperature. Microstructural features were also studied.

2.7. Neighboring or overlapping indentations

In order to study the work hardening of the region surrounding an indentation, the microhardness with 1N load was carried out on S2 and its diagonal was measured. Then indentations with 1N and 0.5N were made very closely to the previous indentation and diagonals of the former and the later were measured. This was repeated with varying distance between the two indentations.

2.8. Flow of material in indentation

The microhardness with 1N load was carried out on all the specimens including the specimen in as received condition. The microstructural features of the flow pattern of the material inside the indentation and consequently the changes in the shape of the indentation were observed immediately after unloading for a certain length of time. Interesting observations were made during this test. Since the diagonal of impression on sintered carbides, even at 1N load was very small, all the observations and measurements were taken at 1000X by replacing the microhardness tester with a higher magnification objective in each case.

III. RESULTS AND DISCUSSION

3.1. Applied load Vs Microhardness

As the applied load is increased, the microhardness value was increasing as shown in Fig.1. This may be attributed to variation in the distribution of carbide particles. It may be noted that, in the load range of 0.8-1N, the variation in microhardness values is comparatively low. This is due to the fact that, since the carbide particles are brittle in nature, they deform elastically and then undergo fracture without any plastic deformation. At very small loads, the area covered under the indenter is very small, hence the number of carbide particles. The resistance offered by the surrounding particles to the relative movement of carbide under indenter may be small, thus resulting in higher diagonal lengths, which inturn resulting in a lower microhardness values. On the other hand, in a fairly high load range, the indenter covers fairly larger area; hence the resistance offered by surrounding particles to the relative movement of carbides under indenter may be high, thus, resulting in lower diagonal lengths and consequently high microhardness values. This observation does not agree with the commonly agreed phenomenon of increasing hardness with decreasing load. However, *Ivan'ko* has proposed a theory regarding the observation of increasing mirohardness with increasing load, in certain cases. *Ivan'ko*'s theory is based on the relative contributions of plastic and elastic deformations in the formation of indentation. Since in sintered carbides in the current investigation, plastic deformation can occur to a limited extent and further the elastic deformation is likely to be small because of its elastic modulus value of 7.07×10^5 N/m^2. There is a wide difference in hardness value between S2&S3 perhaps due to different composition. The results reveal that, the optimum load range for measuring microhardness in sintered carbides is 0.8-1N.

3.2. Effect of rate of loading and unloading on microhardness

No particular trend in microhardness is seen at different loads for different rates of loading and unloading as shown in Fig.2. These irregular variations may be attributed to the variation in the distribution of carbide particles on the matrix. It may be noted that, the effect of these on length of impression diagonal is not significant and no brittle cracking was observed even at the highest rate of loading.

3.3. Indentation Creep

It is observed that, there is no definite relationship existing between the indentation time and length of diagonal as shown in Fig.3. Most of the observations fall within the range of experimental error and largely scatter in observations may be attributed to the non-uniform distribution of the carbides in the matrix. Even the measurement after 1000sec does not show much variation from the average value of the length of diagonal at that particular load of 1N. Therefore, it is impractical to expect any indentation creep at room temperature on sintered carbides.

3.4. Statistical distribution of microhardness

The measured results are arranged in groups. Frequency curves for two similar specimens made by different manufacturers are shown in Fig.4. Though, both the specimens show an almost similar distribution, the variation is significant in each case. The probability plot of ln(n) Vs $(H-H_m)^2$ is drawn to study the nature of distribution, assuming the observations to be normally distributed. The standard deviation for S1 and S2 calculated from the slope of lines in Fig.5 is 22.18 and 22.93 respectively. This scattering may be due to non-uniform distribution of carbides (along with certain size distribution) in the matrix of cobalt. This type of microhardness analysis should prove much useful to control the random errors occurring in the production process. Such a large variation in the microhardness values observed would definitely reflect on the non-uniformity of the performance of cutting edge and therefore, the need for greater control in the uniformity of microhardness values is essential in such products. This wide scattering of the microhardness value may be the source for irregularity seen in the Fig. 2 and Fig.3.

3.5. Effect of heating on microhardness

An average microhardenss of 2.06×10^4 MPa was observed in heated sample S2(a), while unheated specimen S2 showed a mean microhardness of 2.05×10^4 MPa. This indicates that, there is no substantial change in microhardness due to heating. But it was observed that, the oxidation on the specimen occurred upto certain thickness can be removed by polishing. This kind of oxidation, which may occur during usage of sintered carbides may lead to wear of the surface through the inner layers, may not be affected in terms of their microhardeness values.

3.6. Effect of neighboring indentations

Length of indentation values in Table.2 reveals that, there is no occurrence of work hardened region around the indentation. It was ensured by varying the distance of the second indentation from the first one. It could be most simply explained on the basis that, the carbides cannot undergo any amount of plastic deformation and hence work hardening. Only the cobalt can be work hardened, since its amount is only 10%, very little overall work hardening around the indentation was observed.

Table.2. Effect of neighboring indentations in sintered carbides [S2]

(Rate of loading and unloading: 2×10^{-2} N.s^{-1}; Indentation time: 30sec)

Distance between indentations, μm	Length of indentation diagonal, μm		
	1N load	1N load	0.5N load
-	10.064	-	-
0.5920	10.064	9.4456	-
15.392	10.064	9.5048	-
-	9.8272	-	-
0.9472	9.8272	-	9.2352
11.840	9.8272	-	9.3536

3.7. Flow of material in the indentation

In the present investigation, a peculiar feature, indicating certain fringes within the indentation was observed, with all the loads in the range of 0.1-1N, after the removal of load in all the indentations made. Interestingly, the fringe pattern started changing after certain time interval following the load removal through different stages. At each stage, the number of fringes decreased and the convexity of the sides of the indentation were observed, showing curved sides or indentation with what is normally called 'Pincushion' distortion. The time taken for the fringes to completely disappear varied from location to location in the same specimen, ranging from a few seconds to approximately 10min. Since the diagonal is approximately of the order of 10μm, the depth of the indentation is 10/7 = 1.4μm (appx.). Normally four symmetrical fringes are observed, therefore, there are four steps from the surface of the specimen to the center of the indentation and the average step height would be approximately 0.35μm. These observations imply that, there is a flow of material at a very fine scale on the sides of the indentation leading to disappearance of steps of the order of fraction of a micron. The reason for this phenomenon may be explained in the following manner.

In section 3.1, it was assumed that, the carbide particles undergo only relative movement during indentation. After unloading, the carbides tend to move into the indentation with the collapse of sidewalls towards the interior. This is due to the fact that, when the indentation is made, the test specimen experiences large compressive stresses, which results in severe plastic and elastic strain beneath the indenter. These plastic and elastic stains are accommodated completely by the matrix, since carbides are assumed to undergo very little plastic or elastic deformation. It may be understood that, when the indenter is removed, the residual plastic and elastic strains in matrix would tend to recover from the matrix. Since the matrix is only 10% by wt, this recovery process involves considerable shifting of carbide particles. Therefore, step like surfaces are introduced and after certain time when complete recovery has taken place, the curved surfaces result. This inward flow of material is the time dependent process which takes place over few minutes. Therefore, this phenomenon of disappearance of fringes is a stress relaxation process with movement of material. It should be noted that, this process was not observed earlier, because it may be peculiar to a material like sintered carbides with large amount of hard, incompressible particles embedded in a small amount of soft matrix. In order to understand the real nature of this process, it would be worthwhile to search for similar observations on other composite materials with hard particles embedded in a soft matrix. Such studies would be highly valuable in understanding the mechanical behavior of such two phase structures.

IV. CONCLUSIONS

1. It is observed that, increase in applied load results in increase in the micro hardness value in the load range of 0.1-0.8N and the value is almost constant in the load range of 0.8-1N.
2. The effect of rates of loading/unloading on microhardness values is practically negligible.
3. In the load range 0.1-1N, there is no indentation of creep at ambient temperature.
4. A statistical analysis based on microhardness values is suggested to control the random errors occurring in the production process.
5. There is no effect of heating and cooling on microhardness values, except that the oxidation of the surface layers of the specimen occurs upto very small depth.
6. There is no work-hardened region around the indentation.
7. A peculiar feature, showing the fringes inside the indentation suggests that, there is a flow of material into the indentation, which is time dependent. Further work on this phenomenon is required, which could throw light on the mechanical behavior of sintered carbides.

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