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Material Characterization on EN24

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***Abstract*: Ni–Cr–Mo steels such as En24 steel are widely used in machine part members, gears and shafts. En24 steel is generally used in the hardened and tempered condition to achieve an optimum combination of hardness and ductility. In the present study, heat treatment response of En24 steel was investigated by variation of hardening and tempering temperature in relation to microstructure and hardness. The microstructures were studied through a combination of scanning electron microscopy (SEM) and X-ray diffraction (XRD). The specimens tempered at different temperatures (in the range 473–823 K) exhibited decreasing hardness with increase in tempering temperature.**

***Keywords*: Microstructure; hardness; hardening; tempering; abrasion; micro-cutting.**

1. Introduction

Nowadays, the production of cutting tools material with different types of hard coatings is essential for effective machining of hard materials. Cemented carbide tools are most commonly used as a cutting tool material for machining hard materials like alloy steels and high-speed steels, etc. However, cemented carbide tools develop tool wear in a high rate while machining the hard materials. It is generally known that the wear mainly depends on the mechanical, physical and chemical properties of the tool and work piece and the condition at the chip–tool interface. Therefore, the development of suitable coatings is mainly to improve the wear resistance of the cutting tool materials is a major challenge that has been faced by the tool manufacturers. Many monolayer coatings such as TiN, TiAlN, TiC, TiCN and Al2O3 are used as a coating agents on cemented carbide cutting tools, which has improved the hardness on the tool surface and reduced the friction between the tool and the workpiece. As a result, the tool wear has been reduced and the surface finish of the work piece will improve. In the tool geometry, cutting conditions, composition and hardness of the workpiece material are the important factors [1]. Wear by abrasion accounts for more than 50% of the wear related failures. This is not only responsible for material removal, but it also leads to the pre-mature failure in the engineering components [2]. There are several studies based on the effect of heat treatment on microstructure and wear properties of alloying steels [3]-[9] The abrasive wear behavior of the En24 steel has not been studied as a function of heat treatment processes such as hardening and tempering. Similarly, in the past no attempt has been made to correlate dislocation density as a main function of hardening temperature and morphology of the martensitic microstructure. In the present study the microstructural variations that are obtained by heat treatment were systematically investigated by the help of Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) analysis. Similarly, the abrasive wear tests were carried out on En24 steel with different type's microstructures and an attempt that has been made to establish the co-relations.

2. Present work

In the present work, [1] the main expection is the performances of TiAlN, AlCrN and AlCrN/ TiAlN-coated and uncoated tungsten carbide cutting tool inserts which are evaluated by the turning studies that has been conducted on EN24 steel workpiece. The output parameters of the EN24 steel such as cutting forces, tool wear and surface roughness for TiAlN, AlCrN and AlCrN/TiAlN coated carbide cutting tools that are compared with the uncoated carbide tools. As the results show that the AlCrN/ TiAlN coated cutting tool has provided a better surface finish and minimum tool wear rate.

A. En24 properties and experimental needs

EN24 is a very high strength steel alloy which is supplied hardened and tempered. The grade is a Nickel-Chromium-Molybdenum combination, this offers high tensile strength, with good ductility and wear resistance characteristics, with good relative impact properties at low temperature. EN24 is also suitable for a variety of elevated temperature applications. EN24 has a tensile strength of 850-1000 N/mm2. [10]

1) Hardening

Its hardness range is 248-302 HB. The hardening alloy for EN24 is steel due its excellent machinability. The hardening of EN24 is done by uniform heating of the material to 823-850oc until heated through quench in oil.

2) Experimental details

TiAlN, AlCrN and AlCrN (top layer)/TiAlN (bottom layer) bilayer coatings were deposited on the tungsten carbide cutting tool inserts using the physical vapor deposition (cathodic arc vapor deposition) process. Aluminum/ titanium or aluminum/chromium materials were placed as targets on the inner sides of the chamber walls to deposit TiAlN and AlCrN coatings, respectively, onto the tool substrate, and a substrate current of 18.5A was supplied. The hardness and adhesive strength of the coatings are measured using nanoindentor and scratch tester (Ducom model: TR101; India-make), respectively. These confirmed that the developed coatings are better in their characteristics. The machining studies were conducted according to Taguchi's (L9) orthogonal array, using coated and uncoated cemented carbide turning cutting tools on the EN24 alloy steel workpiece material. The hardness of uncoated cemented carbide tool was 23.5 GPa. The hardness and Young's modulus of EN24 alloy steel workpiece are 4.5 GPa and 207*103 N/mm2, respectively.

3) Tempering

Tempering of EN24 is done by heating the material uniformly and through at the selected temperature upto 660oc and hold at that temperature for two hours per inch of total thickness.

4) Chemical composition of en24

The chemical composition of EN24 is shown in the table given below,

Table 1
Chemical composition of EN24

C	Si	Mn	S	P	Cr	Mo	Ni
0.35-0.44	0.10-0.35	0.45-0.70	0.040 max	0.035 max	1-1.4	0.20-0.35	1.3-1.7

5) Applications of en24

EN24 is used in components such as gears, shafts, studs and bolts. It is also used in components subject to high stress and with a large cross section. This includes aircraft, automotive and general engineering, Eg: propeller or gear shafts, connecting rod, aircraft landing gear components.

6) Preparation of titanium carbide

Carbon coating precursors should be firstly synthesized by the help of a sol– gel method. The titanium dioxide (TiO2) sol is to be prepared with the help of tetra butyl titanate, ethanol, acetic acid and D.I. water with the preferred molar ratios of C16H36O4Ti : C2H5OH : CH3COOH : H2O ¼ 1 : 20 : 4 : 6. The tetra butyl titanate was dissolved in ethanol and the acetic acid is added into the solution drop wise with the continuous stirring. Then the mixture of D.I. water and ethanol at the pH value of 3 was dropped into the alkoxide solution. The pH value of the system was maintained between the ranges of 3–3.5 by adding the nitric acid solution. The defined amount of phenol-formaldehyde resin which has a carbon source was dissolved in ethanol and then it is introduced to the system according to the titanium to carbon molar ratio which is been varying from 1 : 1 to 1 : 4. Alternatively, the carbon-coated TiO2 sol solution is to be formed. The gel conditions are obtained after aging at room temperature and it is followed by drying at 1200c to the constant weight and then grinding it into powders. The vacuum condition has to be involved to calcinate the precursors. Approximately 3 grams of precursor powders were pressed and then made them into wafers which is to be at 8 mm in length, 20 mm in diameter under a pressure of 15 MPa. Then, the wafers are to be placed in graphite crucibles at the center of the graphite furnace which is to in vaccum condition. These precursors are to be kept reacted at 14500c for almost 2 h with a constant heating rate of 50^{c} . The pressure that has to be maintained in the vacuum heating process was about 10 Pa.[11]

7) Characterization

Thermo gravimetric analysis is mainly used to determine the carbon yield of the phenol resin and the thermo lysis process of the prepared Titanium Carbide powders. Now take almost 5 mg of the TiC powders and phenol resin were to be heated under air and argon from 250c to 10000c at a heating rate of 100^{C} with an empty Al2O3 crucible. The X-ray diffraction using a Copper target a radiation source of l ¼ 1.540598 °A is operated at 40 kV which was carried out to analyses the phase composition of the produced Titanium Carbide powders. The characteristics of the samples were directly observed with the help of field emission scanning electron microscopy. Samples for the SEM imaging process were fixed on the sample platform with the conductive tape.[11] Transmission electron microscopy must be used to observe characteristics of the prepared particles and to identify their crystal plane by the help of HR-TEM and by the help of selected area electron diffraction (SEAD) images. TEM images were prepared by placing a drop of the suspensions of TiC powders in ethanol over the carbon-coated copper grids. SAED was also recorded with the help of the same equipment.

3. Results and discussion

A. XRD test

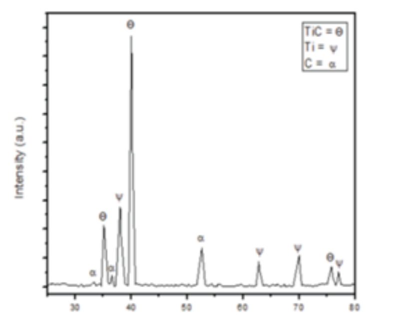

Fig. 1. XRD pattern of titaniumcarbide (TiC)

XRD analysis is mainly based on the constructive interference of the monochromatic X-rays and a crystalline sample. The X-rays are generated by the cathode ray tube and it is filtered to produce the monochromatic radiation to collide, concentrate and it is directed towards the sample. The interactions of the incident rays with the sample produces the constructive interference and a diffracted ray when the conditions satisfies the Bragg's Law ($n\lambda=2d \sin \theta$) which relates the wavelength of the electromagnetic radiation to the diffraction angle and the lattice spacing that are present in a crystalline sample. Titanium carbide is hard material and it is a black color refractory ceramic material having a fcc crystal structure [12], [13]. It is also used for making fuel cell, abrasive material, cutting materials, anti-wear and aerospace materials [14, 15].Titanium carbide (TiC) is one of the most important compounds among the transition metal carbides having properties such as high corrosion resistance and high oxidation,

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high resistance to abrasion and has high resistance to thermal shock [16,17]. It has effective physical and chemical properties such as a high melting temperature (3140 °C), high boiling temperature (4820 °C) , high Vickers hardness (28-35 GPa), high Young's modulus (410-450 GPa), low density (4.93 g/cm–3) and high flexure strength (240-400 N/mm^2) [18,19].

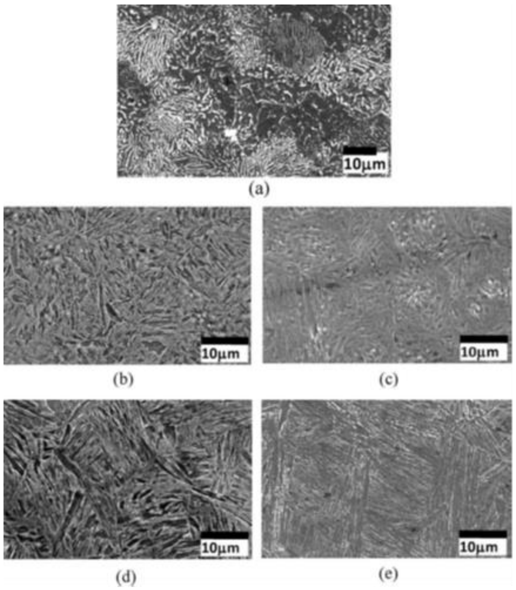

Fig. 2. SEM image of EN24 after annealing

The diffraction patterns from the XRD test confirms the Cubic structure of Titanium carbide (TiC). The calculated average size of the particles is 13 nm.

B. Removing excess carbon

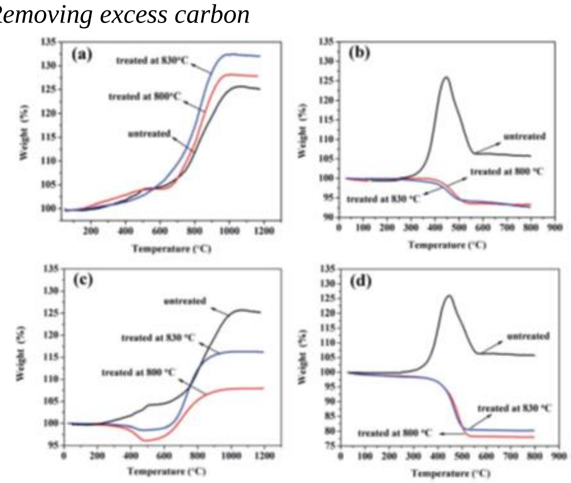

Fig. 3. TGA profiles of TiC-2 (a) and TiC-3 (b) samples treated under hydrogen/argon mixture gas at different temperatures; and TGA profiles of TiC-2 (c) and TiC-3 (d) samples treated under pure hydrogen gas at different temperatures.

Et. al. [11] Excess carbon in the material enables to hinder the particles growth and causes problems to the agglomeration of the produced powders, and it may also covers on the surface of the Titanium Carbide particles and causes an augmentation of the average size. This resulting in reducing its application possibility, specifically in the field of cemented carbide. Loads of surface cleaning processes have been developed and investigated. Catastrophically, high temperature which is required for the removal of amorphous carbon from the surface layer will leads to the decarbonization of TiC and this will affects the performance of TiC powders. In the present work, we have studied that the influence of treatments at different temperatures under both hydrogen/argon (1 : 1) mixture gas and pure hydrogen gas on the prepared TiC-2 and TiC-3 products has been made.

C. UV spectral analysis

Metal/nonmetal doping has a very good influence on light absorption characteristics of TiO2. In the information the comparison of UV spectra between the undoped and the C-doped TiO2 has been prepared at different temperatures for a period of 5 h. The data reveal two prominent features, namely, a red shift of the absorption edge upon C-doping and enhanced absorption in the visible region (400−800 nm). These observations confirm successful doping of carbon into the TiO2 lattice. It has been well-documented that a shift in absorption edge upon doping with carbon introduces a series of localized occupied states in the band gap, and this causes a red shift in the absorption edge into the visible region.[20,21] The long tail in the visible region is attributed to the presence of carbonate species.[21,22,23] It is also worth mentioning that the extent of a red shift in the absorption edge varies with experimental conditions (see Figure S4a and S4b in the Supporting Information). This could be due to the presence of different amounts of dopant, because its quantity depends on the conditions employed during thermal treatment. Furthermore, variation in the intensity of tail that extends into the visible region as a function of time also suggests the presence of varied amounts of carbon/carbonate species. Among the investigated samples, C-TiO2 prepared at 500 °C for 5 h (CT500) seems to have large amounts of carbon dopant, because of its high intensity visible tail. Furthermore, C-TiO2 prepared at 350 °C for 15 h (CT350-15) shows high absorbance in the visible range, compared to samples prepared at the same temperature for long periods of time. This could be due to the release of carbon from the TiO2 lattice during an extended period of heating. The effect of adsorption of analytes on C-TiO2. There is a slight blue shift of ∼6−8 nm in the absorption threshold observed for 4-MBA- and 4-NTPmodified C-TiO2, compared to unmodified C-TiO2.

The magnitude of the shift is larger in the case of the 4-NTPmodified C-TiO2 than that observed for the 4-MBA-modified one. Also, the extent of the blue shift is different for different samples and CT350-15 shows a large shift, compared to other samples prepared at 350 °C for different periods of time. Annealing temperature/time leads to two opposing effects: one leads to an increase in the dopant concentration (C-TiO2) and another leads to loss of carbon from lattice.[26] Coupling between molecule and TiO2 is expected based on the shift in absorption threshold after surface modification. Similar observations have been reported by several other groups.

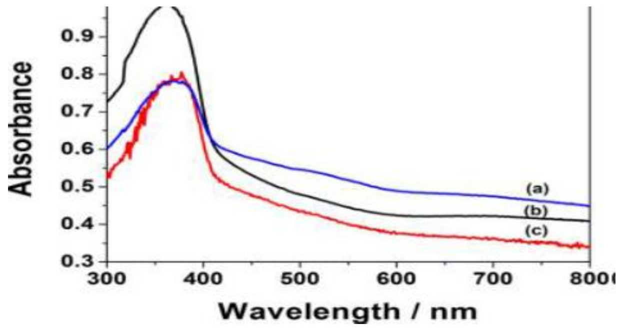

Fig. 4. UV−vis spectra of unmodified C-TiO2 and C-TiO2 modified.

D. Phase characterization of titanium carbide powders

The XRD patterns of samples with various carbon contents heated at 1450oc for 2 h in vacuum atmosphere. It was pronounced that pure titanium carbide formed when the molar ratio of Ti/C ranged from 1: 2 to 1: 4. When in the 1: 1 molar ratio case, the phase of final products was identified to be titanium oxycarbide (TiOxCy), which showed a slightly right shift compared with pure TiC. The preparation of titanium carbide based on the chemical reaction between C and TiO2, as shown by eqn (1) [24]. From the equation, it was clear that the 1:3 molar ratio of Ti/C was needed to form one mole stoichiometric TiC. Therefore, the precursor with 1:1 Ti/C without enough content of carbon made difficulties to obtain pure single-phase titanium carbide. TiO2 + 3C ¼ TiC + 2CO (1) with the increase of carbon content, the reduction process proceeded and the C atoms substituted for O atoms gradually. When the Ti/C ratio dropped to 1: 2, pure titanium carbide appeared. As the content of carbon increased, the higher degree of crystallization of TiC emerged. In our work, the lower carbon content for the appearance of pure titanium carbide might be attributed to the compression molding procedure. The produced reductive gas CO within the compressed precursor could not be taken away immediately, which played an important role as carbon source during the reduction process. Thus, vacuum calcination used here could reduce the use of carbon source compared with those carried out at an atmospheric pressure [25]. The intensity of diffraction peaks for TiC-3 sample was higher compared with that of TiC-2 sample. It could be seen from the patterns that diffraction peaks of TiC-4 matched with the standard spectra of TiC (JCPDS 65-7994), indicating TiC is the predominant composition in TiC-4 sample. However, TiC-4 displays lower and broader diffraction peaks compared with the reference of standard spectra of TiC and other TiC groups (TiC-2 and TiC-3), suggesting some TiCxOy regions between the TiC crystallites. Besides, according to Scherrer formula,[28] TiC-4 samples had the smallest particle size among these TiC powders, which implied that excess carbon could impede the growth of crystallites.[27] This difference in particle size was also demonstrated by the SEM images. The average grain size of products for 1: 2–1: 4 Ti/C molar ratio was calculated by Scherrer formula.

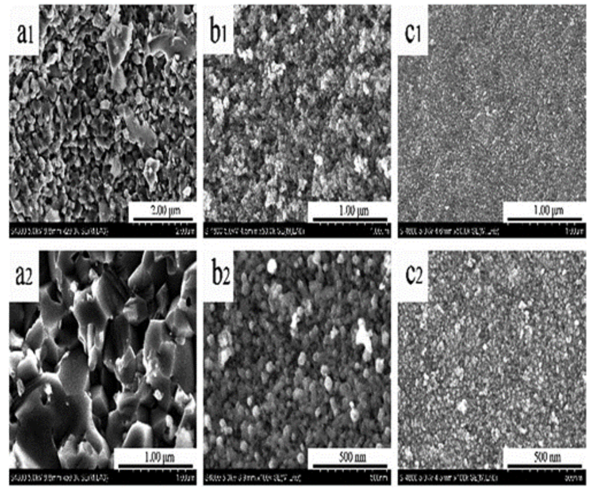

Fig. 5. SEM images of the as-prepared titanium carbide powders with different dosages of carbon: (a1 and a2) TiC-2, (b1 and b2) TiC-3 and (c1 and c2) TiC-4.

4. Conclusion

Thus the coating of TiO2 on the EN steels increases the wear resistance and other material properties. This also increases the microstructural strength. This gives the result better than the other coating process. The solvo thermal synthesis route can be used to synthesize titanium carbide particles. The particles are spherical in shape. [29]-[31]The excessive hydrogen generated during the decomposition of acetone is playing a major role in the conversion of titanium (micro) to titanium nanoparticles. Temperature (800 °C) is appropriate for the reaction but reaction time must be increased to complete the conversion. The thermal efficiency of this EN24 material also determined in this process. Thus the microstructures of En24 steel after hardening and tempering were analyzed using a combination of Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD). The effect of microstructures and hardness on the abrasive wear behavior was also evaluated.

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