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Synthesis and characterization of TiC-reinforced iron-based composites Part II on mechanical characterization

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The hardness, impact toughness and wear resistance properties of Fe-TiC composites, synthesized by aluminothermic reduction of an industrial waste, have been evaluated. The wear resistance property of the composites has been compared with some standard wear resistant materials. It has been found that the wear resistance property of the Fe-TiC composites with mostly pearlitic, fully pearlitic and pearlitic plus cementite type matrix with about 7 to 8 vol% TiC is better than that of a standard high chromium iron. The wear resistance property of ferritic and mostly ferritic type matrix with about 5 vol% TiC is better than that of a standard bearing steel. © *2004 Kluwer Academic Publishers*

1. Introduction

Search for superior wear resistant materials has been alloted a high priority in the field of materials research lately. Although a vaste reserve of wear resistant materials already exists, continuous research activities are on to produce new materials that may replace the existng ones. Composite mterials with steel matrix and ceramic particle reinforcements provide a scope of producing relatively inexpensive wear resistant materials. Iron-based composites with reinforcement of TiC particles have received attention in these class of materials [1–4]. TiC has extremely high hardness (3200 HV) and good thermodynamic stability in Fe alloys [1, 5, 6]. Hence, it has proven its suitability for production of wear-resistant materials [1–4]. A multiphase materials wear reistance property is not dictated by hardness alone. It also depends upon microstructural parameters like volume fraction, size, shape and distribution of embeded particles, properties of matrix and the interfacial bonding between the two phases [4].

Fe-TiC composites have been synthesized by various routes, e.g., powder metallurgy, conventional melting and casting, carbothermic reduction, combustion synthesis, and thermit reduction [7]. Thermit reduction route has advantages over other routes in terms of energy efficiency [7]. The synthesis of Fe-TiC composites with different matrix microstructures and reinforcement volume fractions by aluminothermic reduction of an industrial waste has been described in part I [8]. The scope of the present paper includes evaluation of certain mechanical ptroperties, e.g., hardness, impact toughness and wear resistance of the composites. The wear resistance properties of the composite materials have been evaluated and compared with some standard wear resistant materials like high chromium iron and bearing steel.

2. Experimental procedure

2.1. Synthesis

Fe-TiC composites were synthesized from siliceous sand by aluminothermic reduction method. The details of the synthesis procedure have been described in part I [8].

2.2. Mechanical testing

Various composite materials after processing were subjected to different types of mechanical testing procedures as described below:

2.2.1. Hardness testing

Hardness values of all the composites in as-cast as well as annealed condition were measured in R_c scale using diamond indentor and 150-kg load. The micro-hardness of the individual phases, present in the composites, were determined using Vicker's indentor and 25 gm load. The average of ten measurements has been taken as the hardness of the material/individual phase.

2.2.2. Impact toughness testing

The impact toughness values of a few selected materials were measured in an impact-testing machine. The testings were carried out on some non-standard samples. The samples were machined in the form of unnotched specimens of length of 55 mm and cross section of 10 mm by 5 mm. Pagounis *et al.* [2] used the similar type of specimens for impact toughness measurement of tool steels reinforced with TiC particles which was synthesized by powder metallurgical technique. The average of three measurements has been taken as the impact energy of the material.

2.2.3. Abrasive wear testing

Abrasive wear tests were carried out on 12 mm × 12 mm cross section samples having a thickness of 10 mm, against 220 grit SiC paper affixed to a rotating flat disc of 250 mm diameter [9]. The sliding velocity was fixed at 2.61 ms^{-1} and track diameter was 100 mm. All the experiments were carried out at different loads of 9.8, 14.7, and 19.6 N. Each testing was repeated thrice.

Wear rates of the specimens were computed by the weight loss technique. Prior to weighing, the specimens were cleaned with ethanol to remove the wear debris. Wear data have been plotted as cumulative weight loss per unit area of specimen surface as a function of sliding distance. The abrasive wear behaviour of the composites has been compared with some standard wear resistant materials.

3. Results and discussion

3.1. Hardness

The composites were synthesized with different matrix structures and voume fractions of TiC [8]. Hardness measurements, obtained from the as-cast composites, are presented in Table I. It is observed that the hardness increases from composite T1 to T5. This is due to the change in matrix microstructure as well as the volume fraction of TiC from one composite to another composite. The matrix microstructures of T1, T2, T3, T4 and T5 are ferritic type, ferrito-pearlitic type, mostly pearlitic type, pearlitic type and pearlitic plus cementite type, respectively. The microhardness values of the matrices of the composites have been measured using Vicker's microhardness tester (Table I). It is observed that matrix microhardness increases with the increase in C content as the matrix structure changes from ferritic type in T1 to pearlitic type in T4 and pearlite plus cementite type in T5. In all the cases, the matrix microhardness is higher than the plain carbon steel matrices. This is possibly due to a solid solution strengthening effect of Al and Si as the matrix contains a substantial amount of Al and Si.

The volume fractions of TiC in composites T1, T2, T3, T4, and T5 are 4.5, 5.4, 7.3, 8.5, and 8%, respectively [8]. However, a significant increase in the hardness value of the materials owing to the incorporation of TiC particles is not observed probably due to the nonstoichiometric nature of TiC particles. The microhardness of TiC in the as-cast composites varies from 1800 to 2200 HV. The reported microhardness value of stoichiometric TiC is 2900–3200 HV at 50 g load and 2850–3390 HV at 100 g load [10]. In the present study, hardness of TiC has been measured using Vicker's indentor and 25 g load. A lower load has been used in the present study so that the indentation remains confined well within the particle. The lattice parameter of TiC has been found to be 0.4322 to 0.4320 nm in different composites [11]. The reported lattice parameter of stoichiometric TiC is 0.4327 nm [12]. The microhardness and lattice parameter measurements together with the microstructural changes during annealing (as discussed in Part – I of the paper) establish beyond doubt that TiC particles are non-stoichiometric in nature.

TABLE I Hardness of as-cast composites

Material	Condition	Hardness (R_C)	Micro-hardness of matrix (HV)
T1	as-cast	35	300
T2	as-cast	46	400
T3	as-cast	51	470
T4	as-cast	53	500
T5	as-cast	57	550

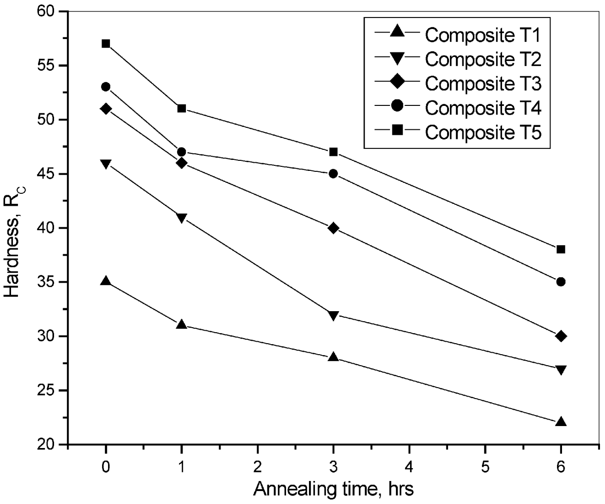

Figure 1 Change in hardness with annealing time for composites T1, T2, T3, T4, and T5 (annealed at 900°C).

As cast composites were subjected to annealing at 900°C for different times (1, 3, and 6 h). Fig. 1 shows the change in hardness with annealing time for composites T1, T2, T3, T4, and T5. It is revealed from the figure that hardness of all the composites decreases with annealing time. This is presumably due to the changes in microstructure. The TiC particles have undergone dissolution during annealing leading to a decrease in the volume fraction of TiC particles in the annealed composite T1. Hence, the hardness of composite T1 decreases during annealing. The hardness of composites T2, T3, T4, and T5 decrease as the matrices of the as-cast composites change from ferrito-pearlitic to ferritic type, mostly pearlitic to ferritic type, pearlitic to ferritic type, and pearlitic plus cementite type to ferritic type, respectivley after annealing. The matrix changes to ferritic type due to diffusion of C from matrix to nonstoichiometric TiC particles.

The composite T4 is subjected to quenching in brine solution. Quenching of the composite T4 increases the hardness from R_C 53 to R_C 64 mainly due to the martensitic transformation (Table II). On tempering, the hardness decreases owing to a relief of quench stress followed by precipitation and coarsening of cementite.

TABLE II Hardness of the quenched and tempered composite T4

Material	Condition	Hardness (R_C)
T4	Quenched	64
T4	Tempered at 200°C for 1/2 h	62
T4	Tempered at 400°C for 1/2 h	56
T4	Tempered at 500°C for 1/2 h	50

TABLE III Impact energy results of as-cast Fe-TiC composites

Material	Impact energy (Joule/cm^2)
T1	25
T3	20
T5	16
Tool steel powder (Fe-0.35%C-4.9%Cr-1.6%Mo-0.6%V, size: <75 μm) + 12 vol% TiC powder (size: 20–30 μm) hipped at 1100°C temperature, 100 MPa pressure and 3 h holding time [4].	13
High-Cr white cast iron powder (Fe-26%Cr-2%C, size: 60% <75 μm + 40% are 75 to 150 μm) + 10 vol% TiC powder (size: average 15 μm) hipped at 1180°C temperature, 100 MPa pressure and 3 h holding time [2].	4 ± 0.5 to 6 ± 0.5 (depending on the heat treatment)

3.2. Impact toughness

One of the applications envisaged in this study is to use the synthesized composite as a cutting tool material. The essential property of a cutting tool material is that the tool material should have high or at least adequate fracture toughness [13]. Hence, impact toughness measurements of few selected composites were carried out at room temperature. The average of three measurements has been taken as the impact energy of the material (Table III). The impact energy value of iron matrix composite, reported in the literature [2, 4], has been included in the Table III for comparison. It is found that the impact toughness values of the composites are low. The low impact toughness of these composites may be due to the fact that the interface region between reinforcement and matrix is very prone to crack nucleation. The crack thus formed propagates along the interface leading to the catastrophic failure of the composite. The impact toughness values are found to decrease from composite T1 to T3. This is possibly due to higher volume fraction of TiC in composite T3 compared to the same in composite T1 since higher is the volume fraction of reinforcement, higher is the interfacial area between matrix and reinforcement. The lower impact toughness of composite T5 compared to T1 or T3 is due to the presence of grain boundary network of cementite as well as higher volume fraction of TiC. Fig. 2 shows a representative factograph of the fracture surface of composite T3. It is observed from the figure that failure occurs by the brittle mode.

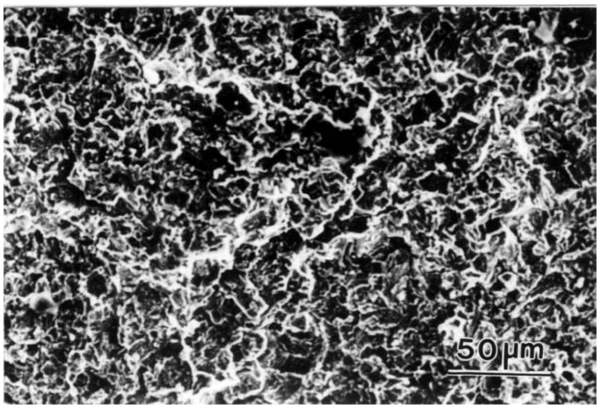

Figure 2 SEM fractograph of composite T3 failed under impact loading.

Although the impact energy values of the synthesized composites are low, the present authors have shown that tools made from composite T5 can machine mild steel rod quite satisfactorily at a cutting velocity of around 35 m/min under cutting fluid application [14].

3.3. Abrasive wear

The main objective in the production of the iron matrix composites is for the use in the applications requiring exceptional wear resistance. The abrasive wear resistance property of the *in-situ* TiC-reinforced iron-based composites were evaluated and compared with standard wear resistant materials.

3.3.1. Comparison of abrasive wear behaviour of as-cast composites (T3, T4, and T5) and a high-chromium iron

The abrasive wear behaviour of as-cast composites T3, T4, and T5 has been compared with a high-chromium iron (Fig. 3). The high-chromium iron contains 2.90% C, 1.00% Si, 1.00% Mn, 0.05% S, 0.032% P, 18.08% Cr, 0.80% Ni, 2.00% Mo, 0.30% Cu, and balance Fe (in wt%) and has a hardness of R_c 60. It is evident from Fig. 3 that wear rate is more in the initial stage and then decreases with the increase in sliding distance for all the materials. Initially the wear of relatively soft matrix controls the wear rate. However after sometime hard carbide particles, exposed to the surface, bear the load resulting a decrease in the wear rate. Decrease in the cutting efficiency of SiC particles owing to blunting by TiC particles is also a contributing factor in lowering the wear rate at the later stage.

Fig. 3 shows that the material loss is more for high-chromium iron than that of the composites T3, T4, and T5. Better wear resistance of the as-cast composites compared to high-chromium iron may be attributed to the following reasons:

(i) High-chromium iron consists of M_7C_3 type carbide having hardness between 1200 to 1500 HV [15].

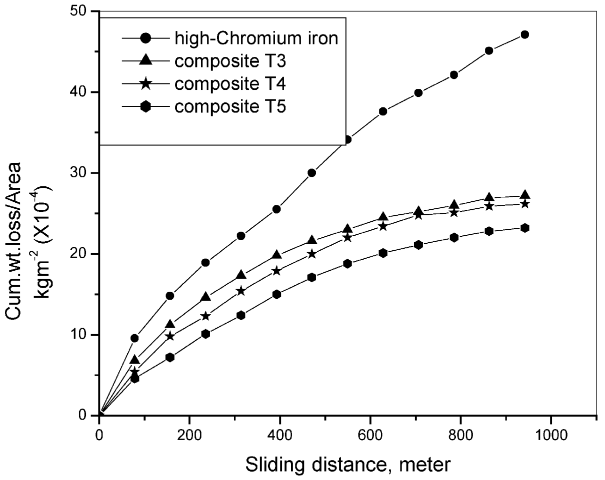

Figure 3 Wear behaviour of as-cast composites T3, T4, T5, and a high-chromium iron at a load of 9.8 N.

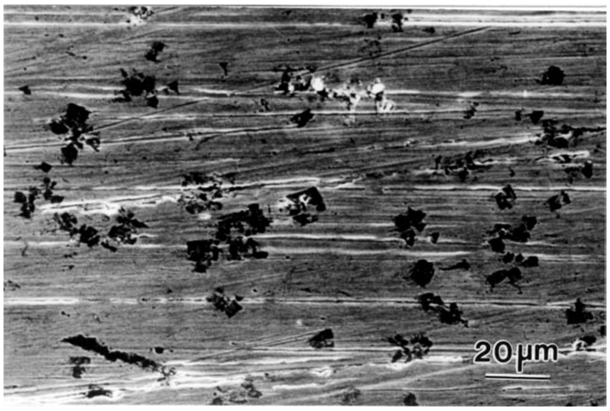

Figure 4 SEM micrograph of Fe-TiC composites showing micro-network of TiC particles.

On the other hand, the hardness of TiC particles in as-cast composites varies between 1800 to 2200 HV. Khrushov *et al.* suggests that higher is the hardness of the reinforcing particle, better is the wear resistance property [16].

(ii) It is found from Fig. 4 that fine (5 μm) TiC particles form micro-networks in the composites that are beneficial by protecting the matrix against wear [4].

(iii) The good bonding between the reinforced TiC particles and the iron matrix can also account for a good wear resistance of the composites. It has been found that no particle spallation has taken place at an applied load of 19.6 N. Micrographs of the worn surfaces will be shown later during the discussion of mechanism of wear. It indicates that matrix can support TiC particles. However, it is reported that martensitic matrix of high-chromium iron cannot support large volume fraction of M_7C_3 type carbide resulting in the spallation of carbide particles, which additionally participate in wear [4].

(iv) It is reported that TiC particles provide composite materials with inherent lubricity and low coefficient of friction [17]. Low coefficient of friction results in low material loss in Fe-TiC composites.

(v) The matrices of the composites contain a substantial amount of Al and Si, which provide oxidation resistance to the matrices and resists oxidative wear.

3.3.2. Effect of load on the wear behaviour of composites T3, T4 and T5, and a high-chromium iron

To find out the effect of load on abrasive wear property, composites T3, T4, and T5 as well as a sample of high-chromium iron were tested at loads of 9.8, 14.7, and 19.6 N. The wear rate has been calculated at a sliding distance of 550 meter for each load and is plotted against load (Fig. 5). It is evident from the figure that wear rate of all the materials increases with the increase in applied load. Abrasive particles cause indentation on the surface of the sample during the course of abrasion. Under an indentation load L, the depth of penetration of the abrasive particles can be written as $x = L\tan\theta/bH_C$ [18], where x = depth of penetration of abrasive particle, b = thickness of substrate and abrasive medium, θ = angle between the substrate and abrasive particle (considering the abrasive particle is triangular in shape with its vertex down) and H_C = hardness of the composite. Higher load causes higher depth of penetration and more material loss. Hence, wear rate increases with an application of higher load. It is also evident from Fig. 5 that wear rates of composites are lower than that of high-chromium iron at all the tested loads. This is possibly due to a good bonding between reinforcing TiC particles and iron matrix.

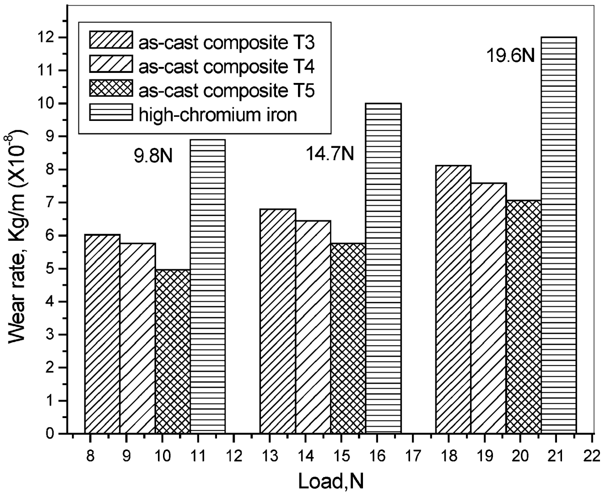

Figure 5 Wear rates of as-cast composites T3, T4, T5, and a high-chromium iron at various loads.

3.3.3. Mechanism of wear

Worn surfaces of the composites T3, T4 and T5 have been studied at different loads (9.8, 14.7 and 19.6 N). Typical SEM micrographs of the worn surfaces of composite T4 at loads of 9.8 N and 19.6 N are shown in Fig. 6a and b. It is evident from Fig. 6 that the material removal occurs by the plowing mechanism. Silicon carbide abrasive particles dig out the matrix and remove material aside. No particle pull out is observed. There is no cracking observed in the reinforcement or at the matrix/reinforcement interface, which suggests strong bonding between the matrix and the reinforcement. No particle gets pulled out from the worn surface even at an applied load of 19.6 N (Fig. 6b). Fig. 7 shows the worn surface of high-chromium iron at a load of 19.6 N. Worn surface shows grooves throughout the surface.

3.3.4. Comparisons of abrasive wear behaviour of as-cast composites T1 and T2, and a 105Cr6 bearing steel

Microstructural observation indicates that composites T1 and T2 consist of ferritic type and mostly ferritic type matrix, respectively, with reinforcement of hard TiC particles [8]. TiC particles have inherent lubricity and low coefficient of friction [17]. The properties of a bearing material include low coefficient of friction, high toughness, and good abrasive wear resistance property.

Composites T1 and T2 have very soft matrix with a hard reinforcement, and hence can be used as bearing materials. The abrasive wear behavior of composites

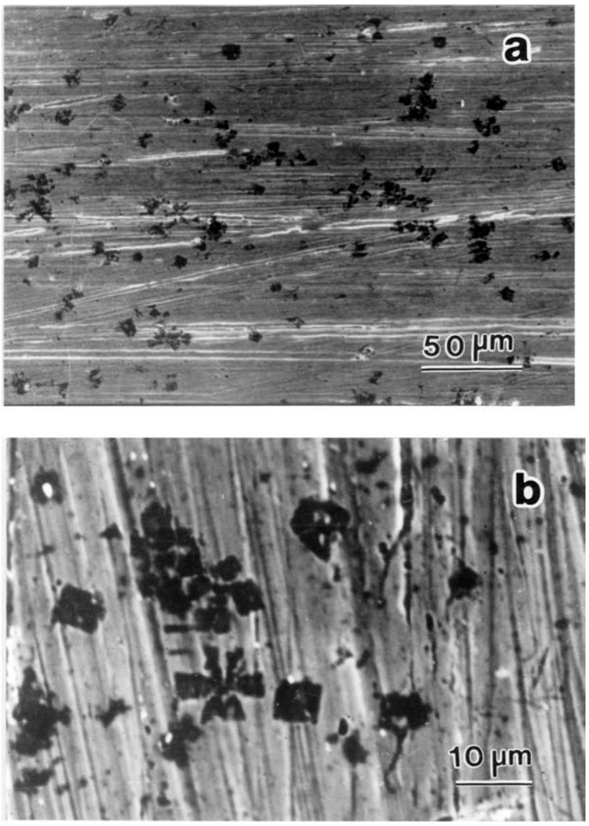

Figure 6 SEM micrographs of the worn surface of as-cast composite T4 at a load of: (a) 9.8 and (b) 19.6 N.

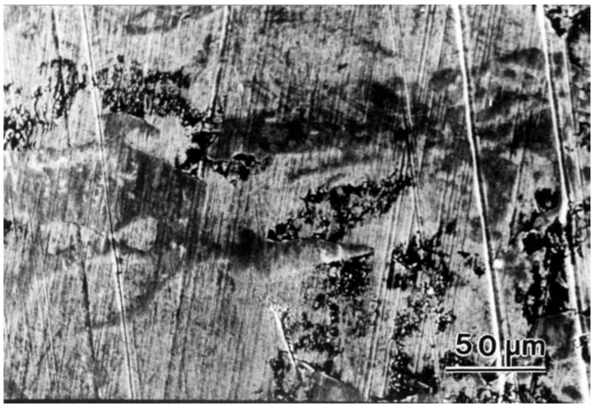

Figure 7 SEM micrograph of the worn surface of high-chromium iron at a load of 19.6 N.

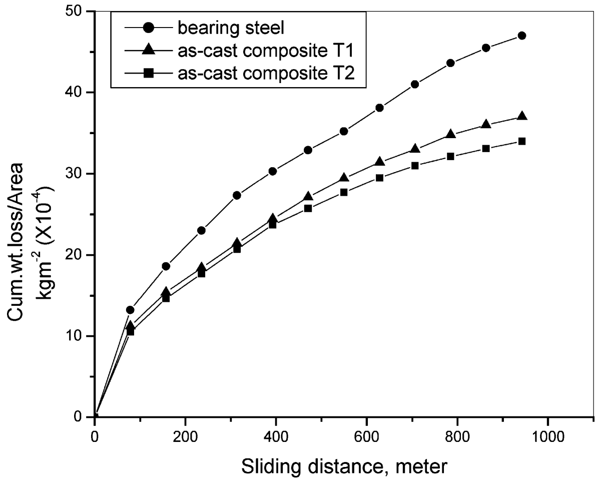

Figure 8 Wear behaviour of as-cast composite T1, T2, and 105Cr6 bearing steel at a load of 9.8 N.

T1 and T2 has been compared with a standard 105Cr6 bearing steel (Fig. 8). The composition of the bearing steel is 1.05% C, 1.52% Cr, 0.5% Mn, 0.33% Si, and balance Fe, (all in weight %) and its hardness is R_C 54. It is evident from Fig. 8 that material loss is more in the case of the bearing steel compared to as-cast composites T1 and T2. This may be due to the re-inforcing effect of hard TiC particles in the composites. TiC particles act as the main load bearing constituents, and resist material loss by abrasion. TiC particles have higher hardness compared to $(Fe,Cr)_3C_2$ present in the bearing steel. The hardness of $(Fe,Cr)_3C_2$ varies from 900 to 1050 HV [19]. Higher is the hardness of the reinforcement, better is the abrasive wear resistance property [16]. Hence, TiC particles can effectively blunt the tip of the abrasive particles and resist abrasive wear.

3.3.5. Effect of quenching and tempering on abrasive wear behavior of as-cast composite T4

In order to assess the effect of quenching treatment on the abrasive wear resistance of *in-situ* composites, the abrasive wear behaviour of as-cast T4, as-quenched T4, and tempered T4 (200°C) has been evaluated at a load of 9.8 N (Fig. 9). It is evident from Fig. 9 that material loss is less in as-quenched T4 compared to as-cast T4. This is owing to the higher hardness of as-quenched T4 compared to as-cast T4. The hardness of as-quenched T4 is R_C 64, whereas the hardness of as-cast T4 is R_C 51. According to standard Archard' s equation (Wear rate $= KLP/H$, where $L =$ sliding distance, $P =$ load, and $H =$ hardness of the material) the wear rate decreases with an increase in hardness [20]. Hence, an as-quenched material has a lower wear rate compared to as-cast composite T4. However, inspite of a lower hardness of tempered-T4 compared to as-quenched T4, the abrasive wear resistance property of tempered-T4 is better than that of as-quenched T4. This is due to the higher toughness of tempered-T4 compared to as-quenched T4. It has been reported that the abrasive wear resistance property increases with the increase of fracture toughness [21].

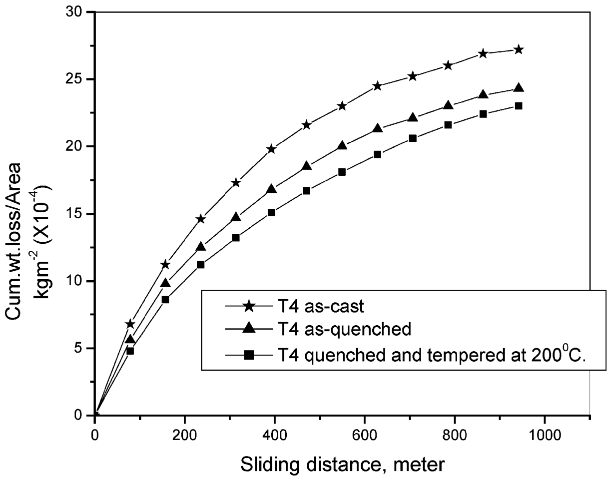

Figure 9 Wear behaviour of as-cast, quenched, and tempered (200°C for 1/2 h) T4 at a load of 9.8 N.

4. Conclusions

1. Hardness of Fe-TiC composites increases due to reinforcement of titanium carbide, but impact toughness decreases. Hardness of the composites does not increase significantly over the unreinforced material inspite of high volume fraction of titanium carbide, as they are non-stoichiometric in nature.

2. The hardness of the Fe-TiC composites with ferritic type matrix decreases during annealing due to the lowering of volume fraction of TiC. Hardness of the Fe-TiC composite with ferrito-pearlitic, pearlitic or pearlitic plus cementite type matrix decreases due to the transformation of these types of matrices to the ferritic type.

3. The composites have an attractive wear resistance property, which exceeds that of standard wear resistant materials. The abrasive wear resistance property of Fe-TiC composites with mostly pearlitic, fully pearlitic, or pearlitic plus cementite type matrix and 7 to 8 vol% TiC is better than that of the high-chromium iron. Abrasive wear resistance property of Fe-TiC composite with mostly ferritic type matrix and about 5 vol% TiC is better than that of the bearing steel.

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