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Performance and limitations of MoS_2/Ti composite coated inserts

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Abstract

Self-lubricating low friction MoS_2/Ti composite coatings were deposited onto hard coated carbide inserts using a hybrid process and were tested for dry high-speed milling and turning of steel. Dry machining is an important objective in industry to reduce environmental and production costs. It was shown that dry machining using MoS_2/Ti composite coatings is possible in some cases. Following an already good knowledge in low speed dry operations including drilling, tapping and threading, the coatings were tested under dry high-speed machining operations (milling and turning) where the temperatures involved are higher. Cutting tool parameters and tool grade and geometry were found to have an influence on the performance of the tools. Temperature and oxidation were investigated separately and correlated to the mechanical, chemical, oxidation and structural behaviour of the tools during machining tests.

Keywords: Dry machining; MoS_2–Ti composite coatings; Hybrid coating; Oxidation

1. Introduction

High speed machining (milling and turning) is recognised as one of the key manufacturing technologies for higher productivity and lower production costs. Existing cutting tools are coated to improve tool life and performance. These coatings are all based on the principle that the coating is much harder than the workpiece material.

In continuous turning operations [1], the temperature of the insert goes up to 700 °C and more. Milling operations are a discontinuous cutting process where the temperatures involved are lower than in turning operations, because the contact time between the insert and the workpiece material is relatively short.

Interrupted cutting results in a considerable variation of the maximum tool temperature of several hundred degrees celsius during single milling cycles, which could result in product accuracy problems. It was thought that a reduction in these problems could be achieved by reducing the heat generation by lowering the friction (very low coefficient of friction) and maintaining good wear resistant properties.

MoS_2/metal composite coatings have been successfully applied to low speed machining operations such as drilling, boring, or tapping [2] where the temperatures involved are much lower, but their suitability for high speed machining operations has not yet been established.

Therefore, the behaviour of MoS_2/Ti composite coated inserts in milling and turning operations were examined.

The objective was to optimise MoS_2/Ti composite coated tools for dry high speed machining of steel. The physical vapour deposition (PVD) magnetron sputtered

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Table 1
Geometry and grade of the PECVD coated inserts used in dry milling and dry turning operation

	Type	Geometry	Grade	Hard Coating
Turning operation	1	SNUN120408	TN250	TiN-TiCN-TiN[a]
	2	CNMA120408	TN7010	TiN-TiCN-Al2O3-ZrCN[a]
Milling operation	3	SEKN1203AF.N-1	TPC25	TiN-TiCN-TiN[a]
	4	SEKR1203AFN-MS	TPC35	TiN-TiCN-TiN[a]

[a] Blasted.

MoS_2/Ti composite coatings were optimised with respect to the hard base layers deposited by plasma-enhanced chemical vapour deposition (PECVD) processes (chemical composition, thickness…). PECVD TiN-coated cemented carbides are used in many metal cutting applications, especially in milling, drilling, threading, but also in turning operations [3,4].

2. Experimental

2.1. Substrates and tools

Selections of four inserts from 15 different geometries and grades of carbides inserts and PECVD hard layers were made for turning and milling operations. In turning operations, geometries and grade of inserts type 1 and 2 (Table 1) were used as they gave the highest tool life during the standard turning operation (Table 1) and were used as a reference for the study. In milling operations, however, geometries and grades of inserts type 3 and type 4 (Table 1) were used for the same reason as mentioned above.

It has been reported that surface roughness has a critical effect on MoS_2/Ti composite coatings friction-wear properties as the coatings are only 1 μm thick. Moreover, the design of sequential multi-technology coating processes is not simple as hard PECVD coatings have tensile stresses and hard PVD coatings have compressive stresses. A compromise solution was developed by inducing a superficial compressive stress via micro-blasting of the PECVD coatings, which was compatible with the low compressive stress of MoS_2/Ti composite coating (low compressive stress of 0.5 GPa in comparison to PVD TiN). Micro-blasted samples also have a better surface finish in comparison to coated ones.

MoS_2/Ti composite coatings were deposited directly on AISI 316L thin foils and plates and on top of PECVD coated turning inserts using a Teer coatings UDP closed field unbalanced magnetron sputter ion plating system (CFUMSPIP) [5,6]. Coatings were deposited with various Ti contents achieved by applying different power on the target.

The structure of the MoS_2/Ti composite coatings were analysed by X-ray diffraction (XRD). XRD measurements were performed using a Philips X-ray X'Pert diffractometer equipment with Cu-Kα radiation source (0.154046 and 1.54439 nm), a Ni filter and a goniomter PW3020 (θ–2θ geometry, 3–100° 2θ range, scan speed of 0.5° min^{-1}). Measurements were taken only between 10 and 70°. Composition and surface morphology were analysed by scanning electron microscopy (SEM) fitted with EDAX.

Mechanical properties such as adhesion, plastic hardness and elastic modulus, residual stress and critical fracture strain of bending of a thin foil for intrinsic stress determination were also measured.

In addition, the temperature effect on the structure and mechanical properties of MoS_2/Ti composite coatings was studied.

Machining tests with these conventional tools (Table 1) were carried out on workpiece material 34CrNiMo6 (annealed condition, with a tensile strength of 800–950 N/mm^2 with an average hardness of 21–25 Hrc).

- Adhesion strength was investigated by scratch tests (see Renevier et al. [7] for details) while increasing the load from 10 to 60 N (PECVD)-80 N (MoST).
- Friction-Wear tests were performed (see Renevier et al. [7] for details) using the same instrument at 50 N load for 2000 cycles.
- Hardness–elastic modulus of the coating were measured by a nano-indentation system. The indentation hysteresis curves were analysed following the procedure proposed by Oliver and Pharr [8].
- Structure of the coating was analysed by X-ray diffraction (XRD) method using Cu-Kα radiation.
- Concentration of Ti, Mo, S in the coating was examined by energy dispersive spectroscopy (EDS).
- Morphology was studied by scanning electron microscopy (SEM).
- Residual stresses were evaluated by the thin foil method. For this method, the radius of curvature of a thin strip of coated material was measured. For the residual stress evaluation, the Stoney formula was used [9]:

$$\sigma_{\text{res}} = \frac{E_s d_s^2}{6R(1-\nu_s)d_c}$$

In this equation, σ_{res} (GPa) is the residual stress, d (m) is the thickness, E_s (GPa) is the elastic modulus, ν is Poisson's ratio, and R (m) is the curvature of the foil.

Table 2
The cutting conditions for turning operation

Parameter	Value
Rake angle	6° negative
Cutting speed	$v_c = 150–250$ m/min
Feed rate	$f = 0.125–0.315$ mm/rev
Depth of cut	$a_p = 1.5–2.5$ mm
Cutting fluid	None
Standard condition	$v_c = 250$ m/min $f = 0.315$ mm/rev $a_p = 2.5$ mm

The subscripts s and c are used for the substrate and coating, respectively.

- The coatings were mechanically characterised by bulging and bending in tension a thin foil for the intrinsic stress determination. The point of failure was determined by using an electrochemical method. In this method, the sample was kept at a constant electrochemical potential (using the standard calomel electrode). When cracks are formed in the coating, the electrolyte comes into contact with the stainless steel. Because stainless steel has a different corrosion potential compared to the coating, the current of corrosion dramatically changes. This change in current of corrosion was taken to be the point of failure.

2.2. Turning operation

The cutting conditions are summarised in Table 2.

2.3. Milling operation

The cutting conditions are summarised in Table 3. The tool life obtained with a flank wear threshold of $VB_B \leq 0.4$ mm is used as a criterion for a comparative evaluation of the coating performance. The quantity VB_B was measured by use of an optical measurement system.

Table 3
The cutting conditions for milling operation

Parameter	Value
Cutting speed	$v_c = 125–250$ m/min
Feed rate	$F_z = 0.25$ mm/rev.tooth
Depth of cut	$a_p = 1.5$ mm
Rotational diameter	$d = 80$ mm
Cutting fluid	None
Number of inserts used in toolholder (+dummies)	1
Standard condition	$v_c = 250$ m/min $F_z = 0.25$ mm/rev.tooth $a_p = 1.5$ mm

3. Results and discussion

3.1. Fundamental structure and mechanical properties of the MoS_2/Ti composite coatings

MoS_2/Ti composite coatings show different mechanical properties as the Ti concentration in the coating increases. Therefore, it is very important to know the relation between the chemical concentration of the coating and its effects on the mechanical properties. XRD patterns were reported in [2,6,7] with respect to the Ti concentration showing that Ti addition (and other metals reported in Renevier et al. [7]) prohibits the formation of a micro-crystallised structure. Two forms of MoST

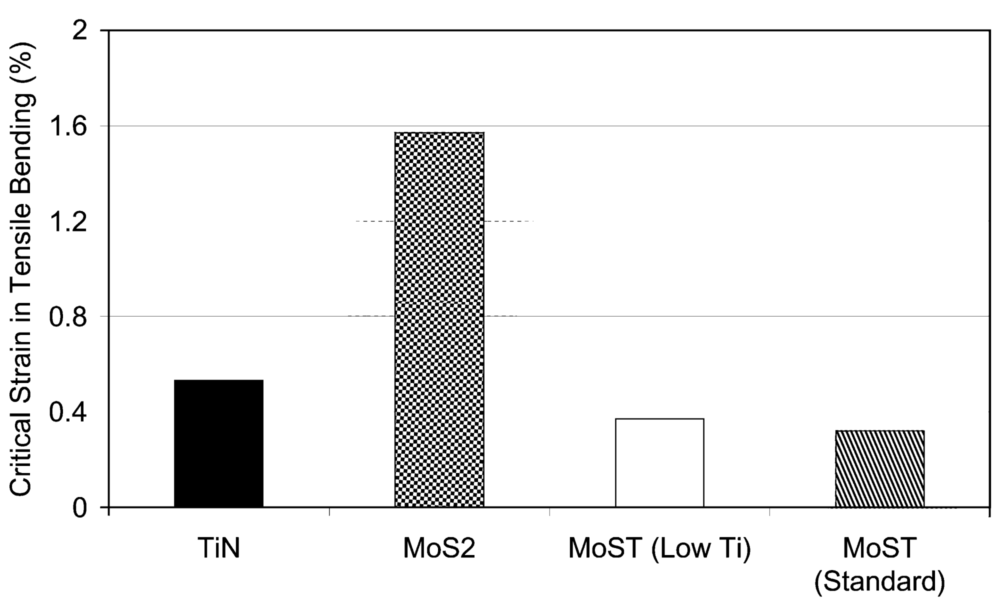

Fig. 1. Comparison of critical strain of TiN, MoS_2 and MoST coatings on SS316 stainless steel as a function of Ti concentration.

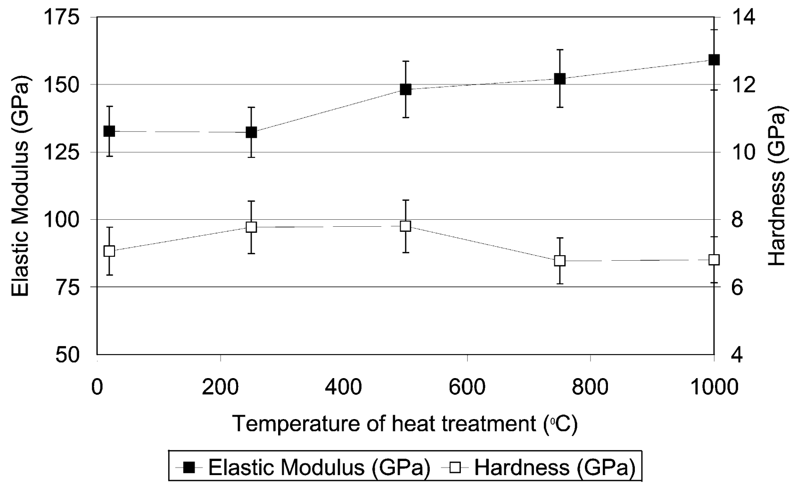

Fig. 2. Dependence of the elastic modulus and the hardness of MoS_2/Ti composite coatings [$X(Ti) = 16$ at.%] on the heat treatment temperature in vacuum.

were studied in more detail: these are MoS_2/Ti composite coatings reported before 1998 (low titanium content, 5–10 at.%) and MoS_2/Ti composite coatings reported after 1998 (standard, 10–20 at.%) [6].

The hardness and elastic modulus of MoS_2 coatings were approximately 5 and 110 GPa, respectively. For MoS_2/Ti composite coatings, however, the hardness and elastic modulus increased to 7 and 130 GPa, respectively, as the Ti concentration increased. It was impossible to find a chipped area within the scratched channel following scratch testing up to 80 N, so it was concluded that the critical load for MoS_2/Ti composite coatings was above 80 N.

Residual stresses of the coatings were always compressive. It was clear that residual stresses of MoS_2/Ti composite coatings dramatically increase by adding Ti to MoS_2.

The critical strain to initiate cracks in MoS_2/Ti composite coatings by bending was only 0.3% and was much higher for pure MoS_2 coatings (1.6%), as shown in Fig. 1. Generally, thin coatings are designed to start cracking without plastic deformation, in tensile bending conditions. However, the extremely high critical strain of MoS_2 coating implies that this coating was plastically deformed before cracking.

3.2. The change of structure and mechanical properties of MoS_2/Ti composite coatings by heat-treatment in vacuum and in air (mechanism of oxidation of the coating)

The main objective of this study was the application of MoS_2/Ti composite coatings to high-speed dry machining operations. In high-speed dry machining conditions, the temperature goes up to 700 °C or more. If the temperature exceeds 500 °C, most standard wear resistant coatings were found to be in an oxidised state. Therefore, investigation of the mechanism of crystallisation and oxidation of the coatings is essential. It was important to separate the effect of heat (vacuum heat treatment) from the effect of oxidation (heat treatment in air). MoS_2 coatings are used as a solid lubricant coating in vacuum conditions and are known to be stable at rather high temperature. A heat-treatment under vacuum was performed as reference to assess any change of structure or mechanical properties of the MoS_2/Ti composite coatings.

Fig. 2 shows the dependence of elastic modulus and hardness of MoS_2/Ti composite (standard) coatings with heat treatment temperature. The hardness was increased

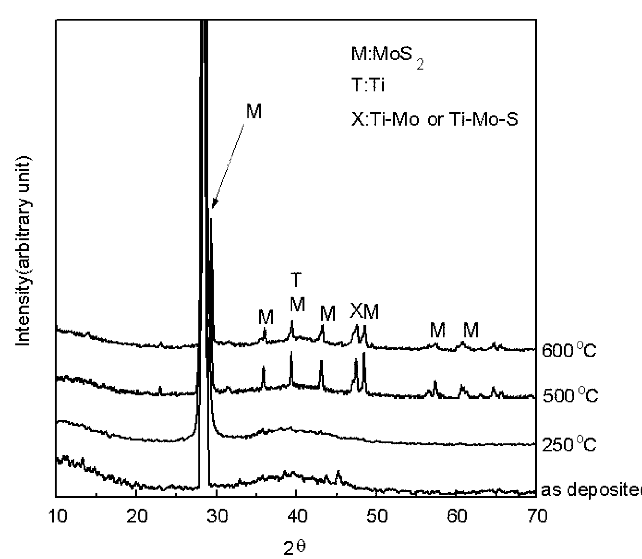

Fig. 3. XRD patterns for MoS_2/Ti composite coatings [$X(Ti) = 16$ at.%] after heat treatment for 1 h in vacuum.

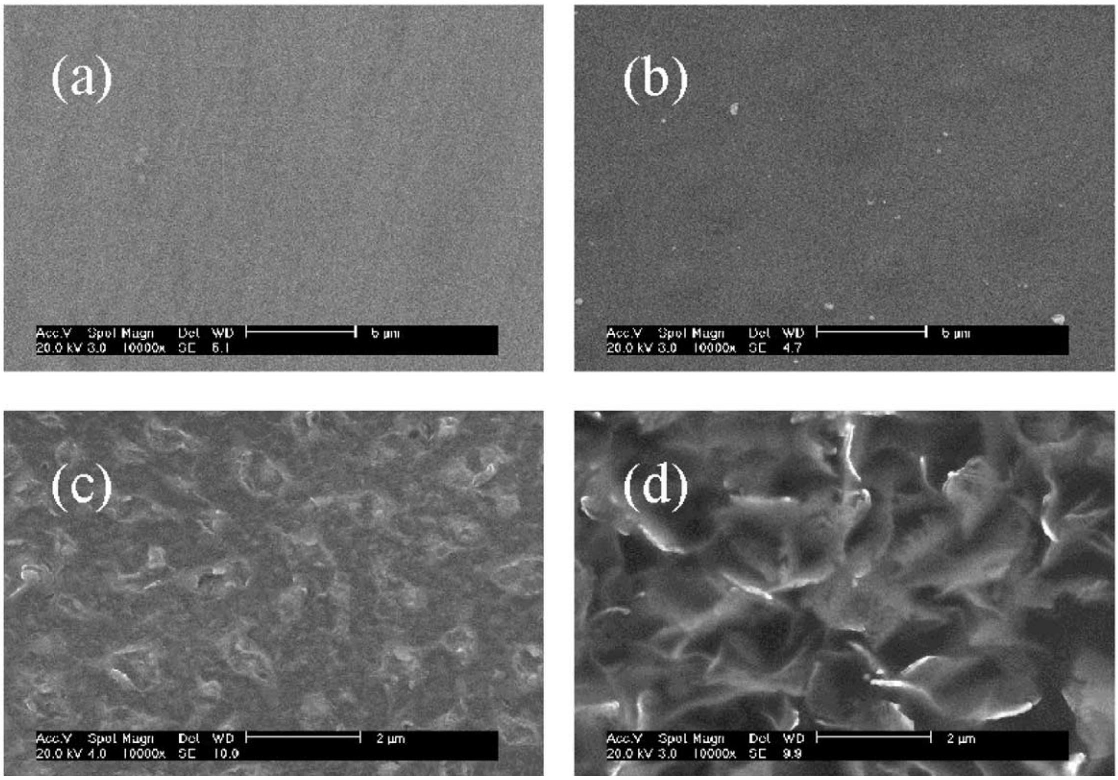

Fig. 4. Surface morphology of MoS_2/Ti composite coatings [X(Ti) = 16 at.%] after heat treatment for 1 h in vacuum. (a) As deposited; (b) at 250 °C; (c) at 500 °C; (d) at 750 °C.

by vacuum heat-treatment and then decreased again if the heat-treatment temperature is above 600 °C. Fig. 3 shows the corresponding XRD patterns for heat-treated MoS_2/Ti composite coatings. This figure shows that MoS_2/Ti composite coatings micro- or macro-crystallise and Ti is segregated from the coating if the heat-treatment temperature is higher than 500 °C. This effect can be related to the decrease in coating hardness.

Fig. 4 shows the surface morphologies of the coatings after vacuum heat-treatment. The surface becomes very rough if the treated temperature exceeds 500 °C, which might result in an increase in friction coefficient and a change in other mechanical properties. The concentration of sulfur (S) abruptly decreased when the temperature reached 750 °C (Fig. 5). This implies the decomposition of the coatings above 750 °C.

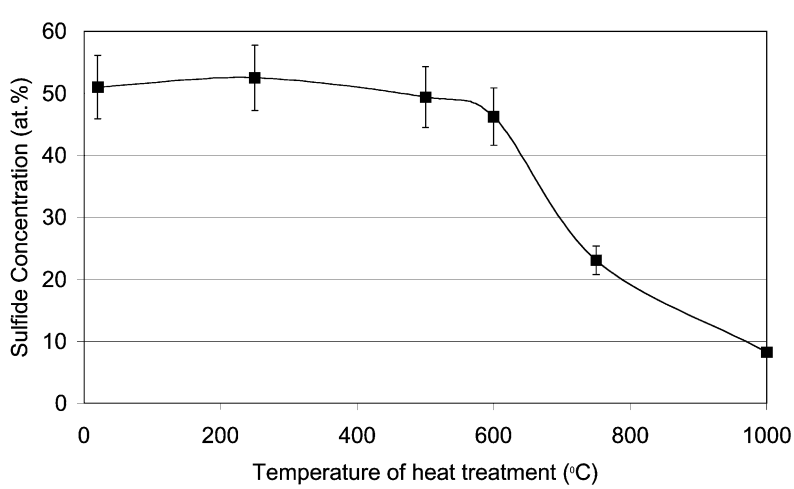

Fig. 5. Dependence of the S concentration of MoS_2/Ti composite coating [X(Ti) = 16 at.%] for as deposited state) on the heat treatment temperature after heat treatment for 1 h in vacuum.

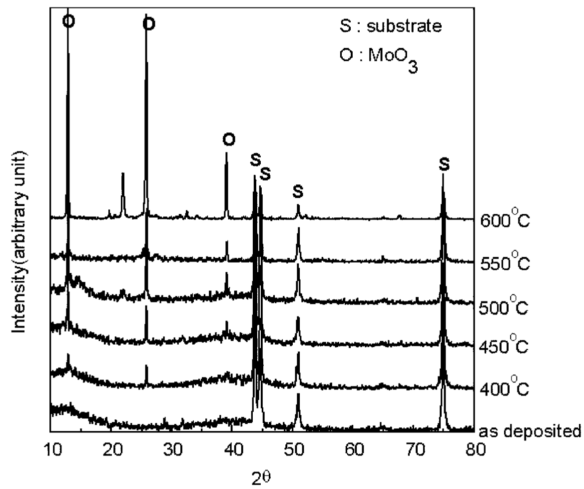

Fig. 6. XRD patterns for MoS_2/Ti composite coatings after oxidation for 10 min in air.

The as deposited coating is not fully dense. When the temperature increases, the lattice parameter of MoS_2/Ti increases slightly, this can explain the increase of hardness in the coating. When the temperature increases further (crystal growth), the lattice parameter cannot expand, there is phase separation (see XRD and SEM) The residues obtained then have a lower hardness.

The XRD patterns (Fig. 6) of MoS_2/Ti composite (standard) coatings after oxidation in air for 10 min at various temperatures show that the coating started to be superficially oxidised above 400 °C (the top 50 nm consists of pure MoS_2 for colour which fully oxidised at 350 °C) and were fully oxidised when the temperature had reached 500 °C.

The surface morphology observation of the coatings after oxidation in air (Fig. 7) shows the presence of many crystallites on the oxidised surface as the temperature increases, and the coatings were completely delaminated above 600 °C. Above 500 °C (Fig. 8) the concentration of sulfur (*S*) decreases dramatically. In addition, the crystallites of the oxidised surface contained a higher fraction of Mo than the matrix of the coating (Fig. 9 and Fig. 10). Above 450 °C (Fig. 8), the adhesion strength decreased dramatically with the temperature due to oxidation, which resulted in lowering the wear resistance. Therefore, it was concluded from the results that the mechanical properties of MoS_2/Ti composite coatings degrade by oxidation for temperatures above 450 °C.

3.3. Machining experiments with composite coated tools

3.3.1. Turning test

During the first 30 s of turning, MoS_2/Ti coating is chipped off from the contact zone of the inserts. This phenomenon was observed for most inserts irrespective of the Titanium concentration in the coating or machining operation (see for example dot mapping [10]). The worn area grows at a constant rate, and the wear depths of each insert are plotted against cutting time.

The flank wear of the coated inserts was not affected by the MoS_2/Ti coatings, whereas the grade and geometry of the PECVD coated inserts has a major impact

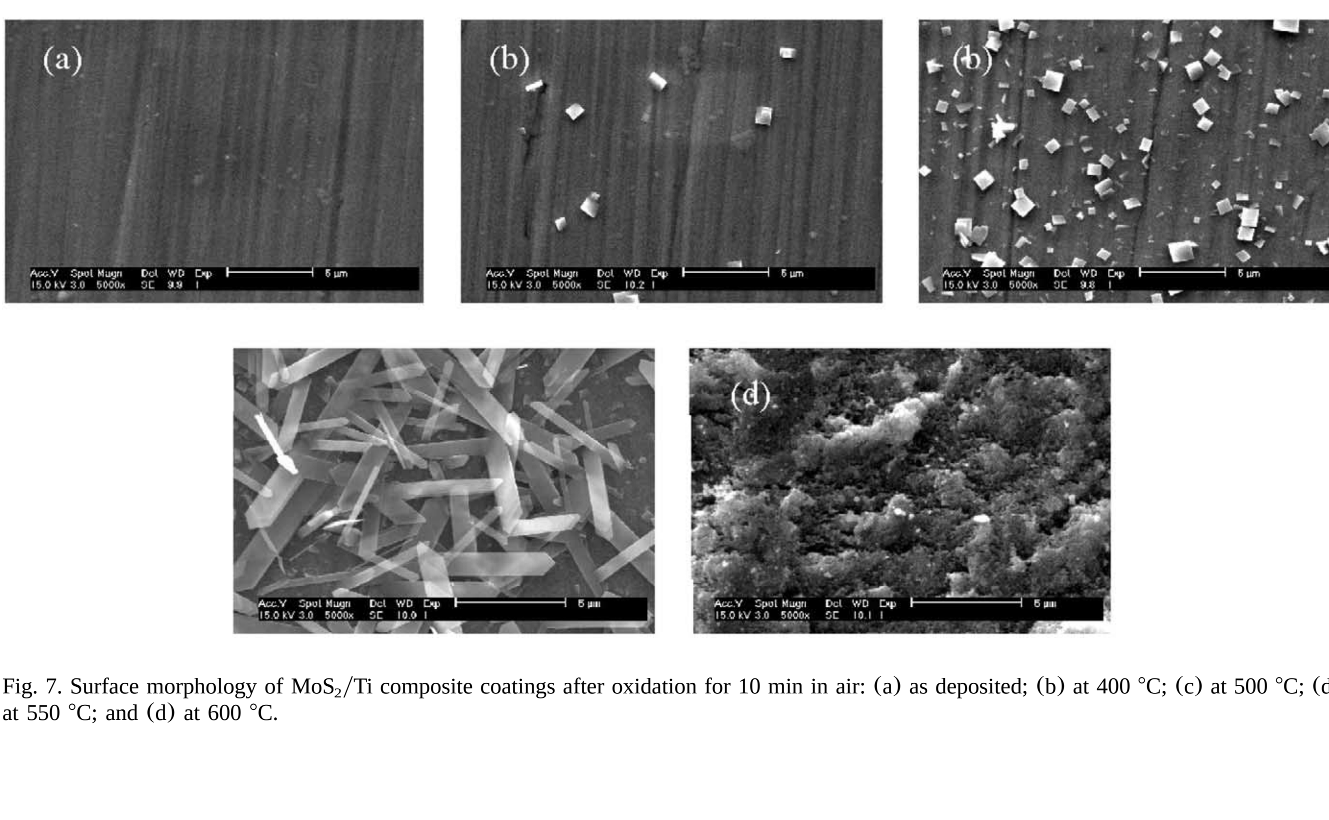

Fig. 7. Surface morphology of MoS_2/Ti composite coatings after oxidation for 10 min in air: (a) as deposited; (b) at 400 °C; (c) at 500 °C; (d) at 550 °C; and (d) at 600 °C.

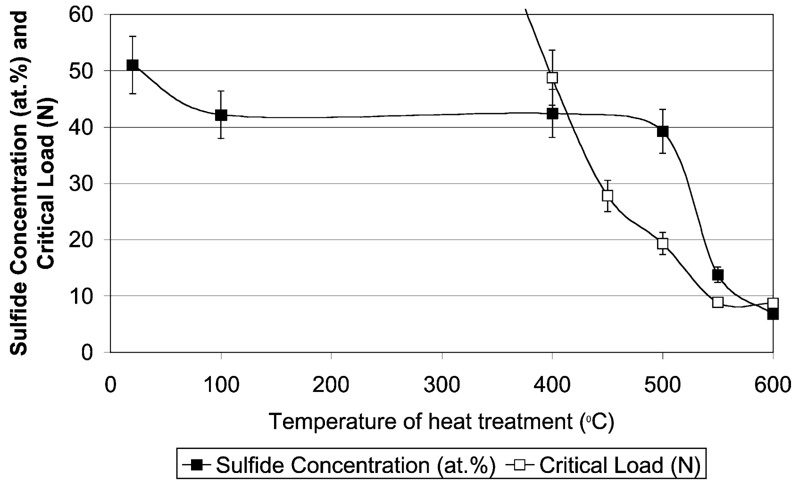

Fig. 8. Dependence of the: (a) S concentration; and (b) the adhesion strength of MoST coating on the oxidation temperature after 10 min oxidation in air.

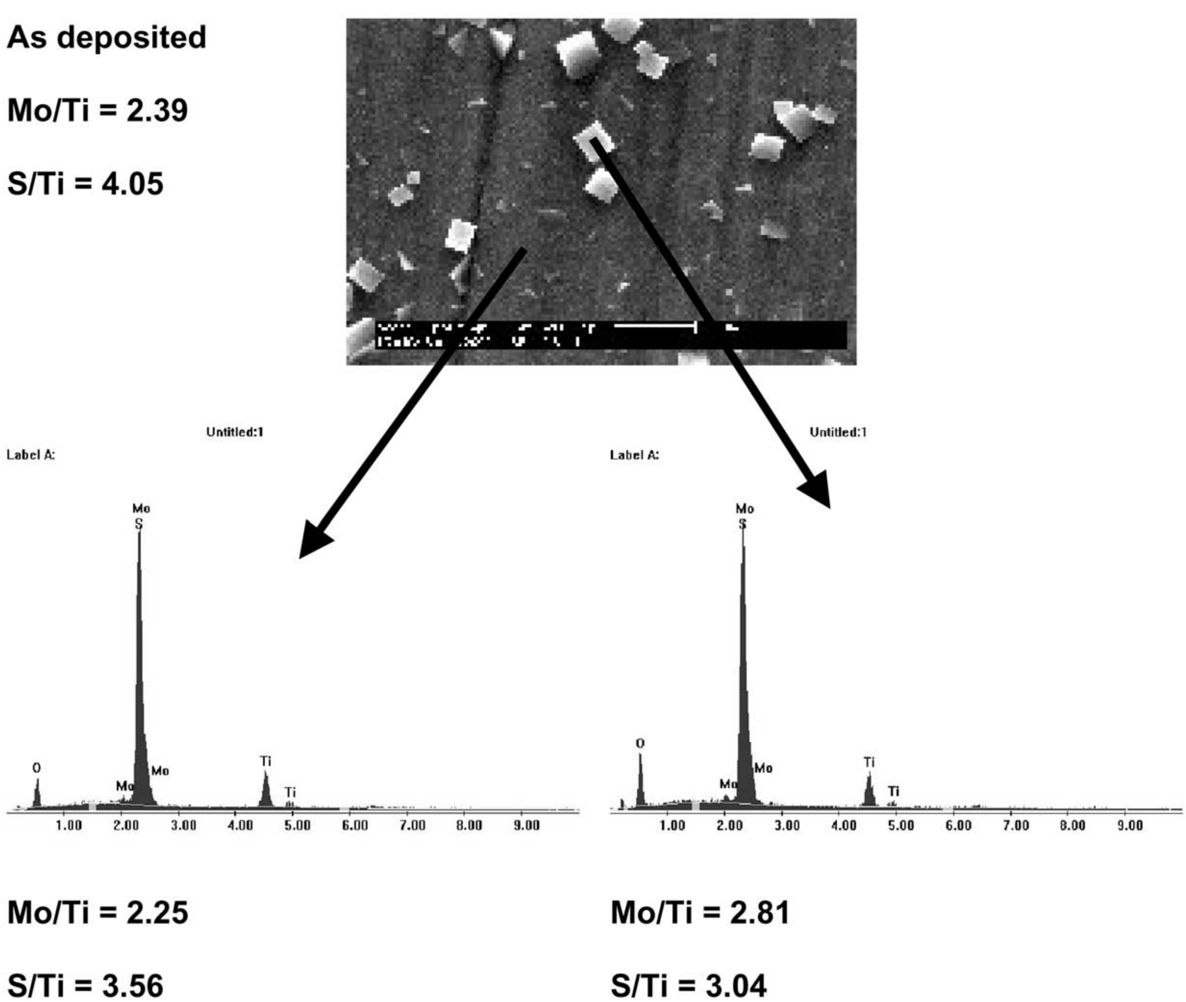

Fig. 9. Surface morphologies and their composition of MoS_2/Ti composite coated insert which was oxidised at 500 °C for 10 min in air.

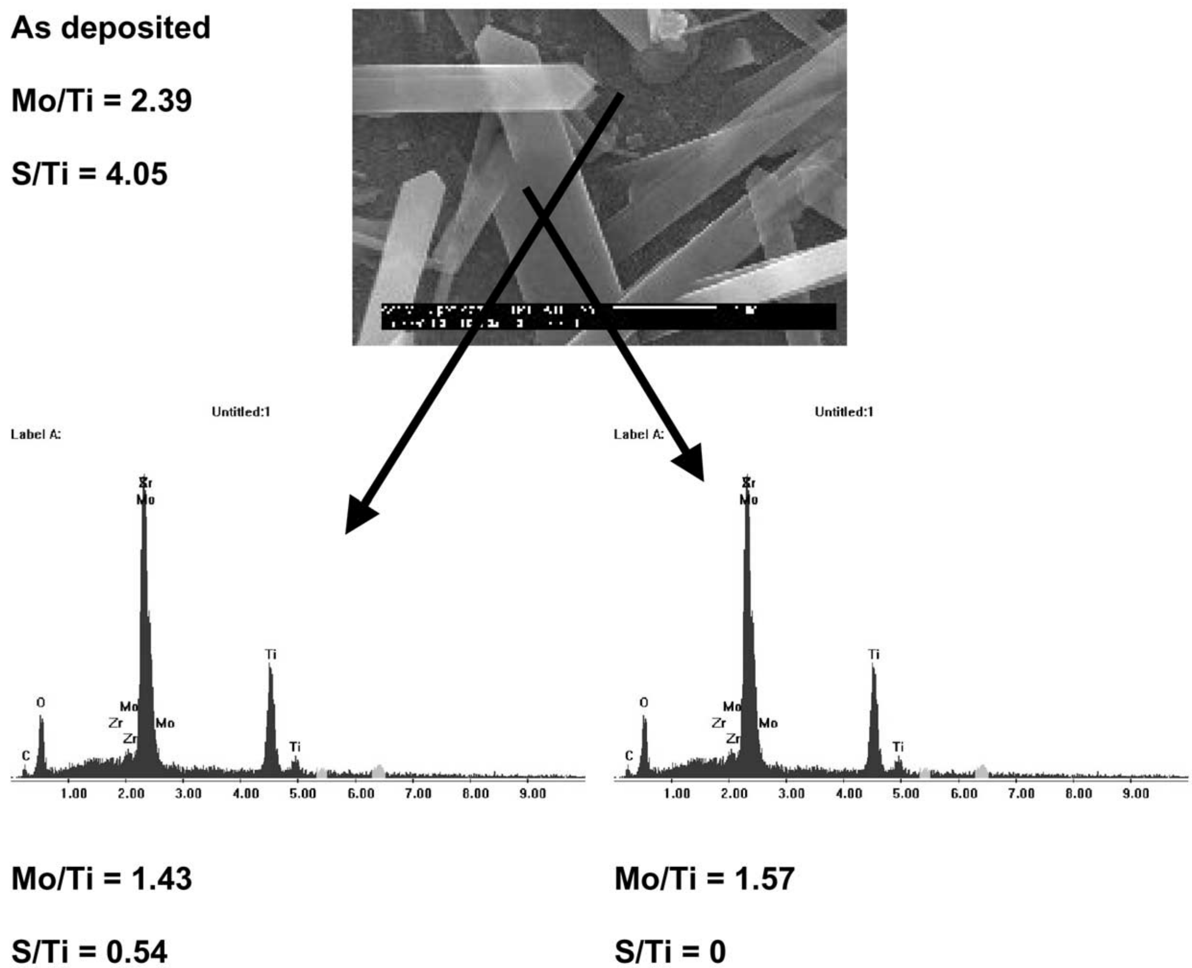

Fig. 10. Surface morphologies and their composition of MoS_2/Ti composite coating which was oxidised at 550 °C for 10 min in air.

on the temperature of chips and insert during the continuous operation.

From examination of the surface morphology of the coated insert (Fig. 7 and Fig. 11) a similar pattern can be observed for the as deposited coated inserts and the tested insert far from the contact zone with the work-piece material, whereas the coated insert heat treated in air for 1 h at 550–600 °C shows a similar pattern to the tested insert (Fig. 7 and Fig. 11)

Therefore, cutting parameters such as cutting speed, feed rate and depth of cut were changed to reduce the temperature dissipated in the tools during the experiment. However, by changing the feed rate, it was not possible to find acceptable parameters for continuous turning operations where MoS_2/Ti composite coatings could have a beneficial effect (Fig. 12). Consequently, MoS_2/Ti composite coatings are not suitable for long contact interval continuous turning using type 1 and type 2 inserts. Other geometry and grades could be investigated as well as machining parameters.

3.3.2. Milling test

In Fig. 13, the tool life of each insert is summarised for each cutting condition. For MoS_2 coatings which are already developed as a solid lubricant coating and are well applied to low speed machining operations such as drilling [11], the tool life of the insert with MoS_2 coating is exactly the same as that without MoS_2 coating. It is suggested that the high temperature during the operation makes it difficult to apply this coating to the high speed machining operation. This implies that MoS_2 coatings can never be applied for high speed machining operation. However, the tool life of the insert with MoS_2/Ti composite coatings was much higher than that without MoS_2/Ti composite coatings. The tool life of the insert with MoS_2/Ti composite (low Ti) coatings decreased much faster than that with MoS_2/Ti composite (standard) coatings, as the cutting speed increased. This difference is supposed to result from the oxidation resistance, that is, the oxidation resistance of MoS_2/Ti

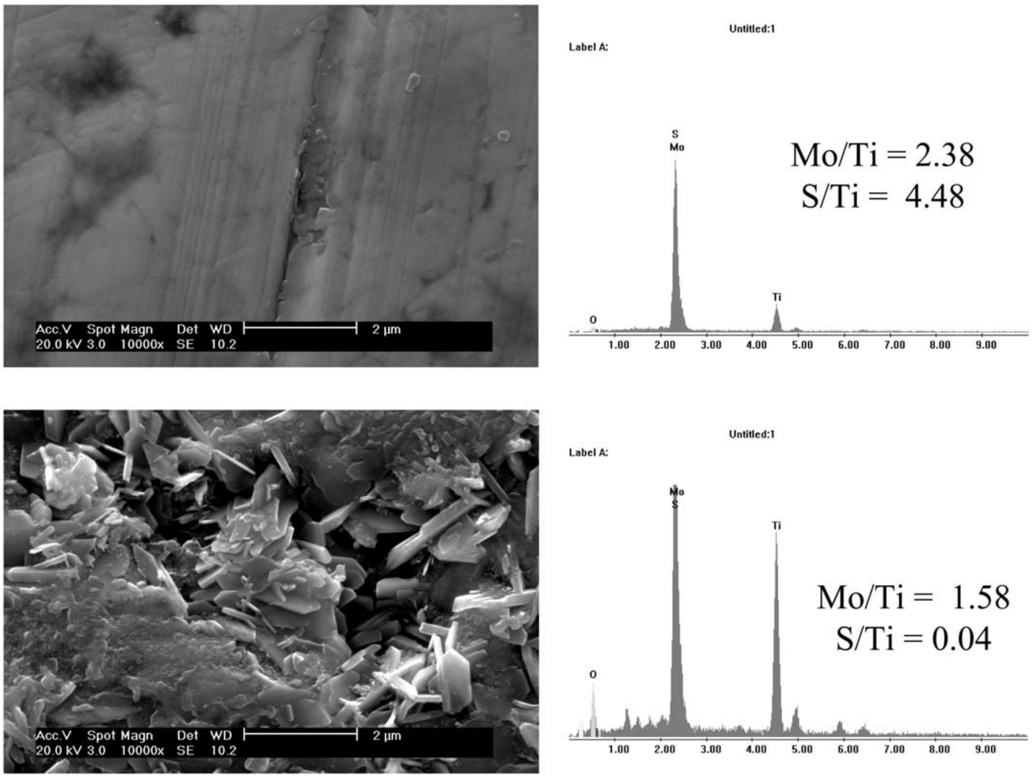

Fig. 11. Surface morphology and its composition of rake surface of MoST coated turning insert (standard condition, after 1 min turning test, type 1). (a) Far from the contact zone with workpiece material and (b) underneath the contact zone with workpiece material.

composite (standard) coatings is much higher than that of MoS_2/Ti composite (low Ti) coatings.

The suitability of MoS_2/Ti composite coatings for milling and turning operations is summarised in Table 4.

4. Conclusions

MoST coatings were coated on stainless steels and (milling and turning) inserts using a Teer Coatings UDP magnetron sputter ion plating system. The fundamental

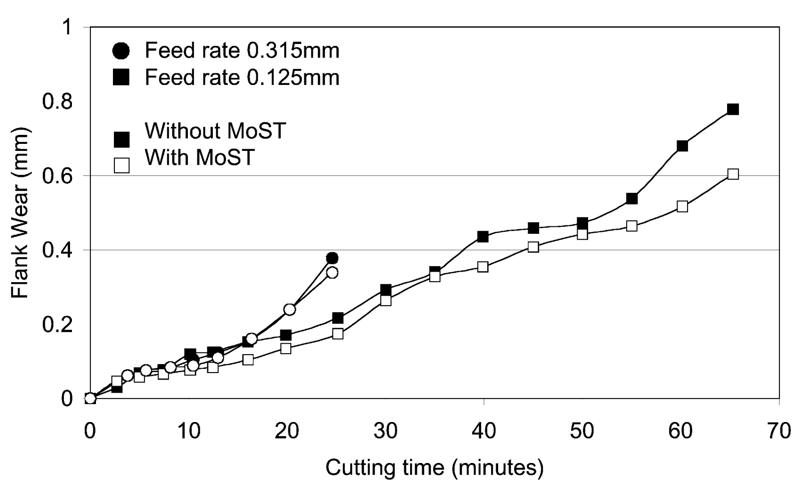

Fig. 12. Summary of dependence of the flank wear of turning inserts on the time of cutting in continuous turning conditions. (type 2) for two different feed rates.

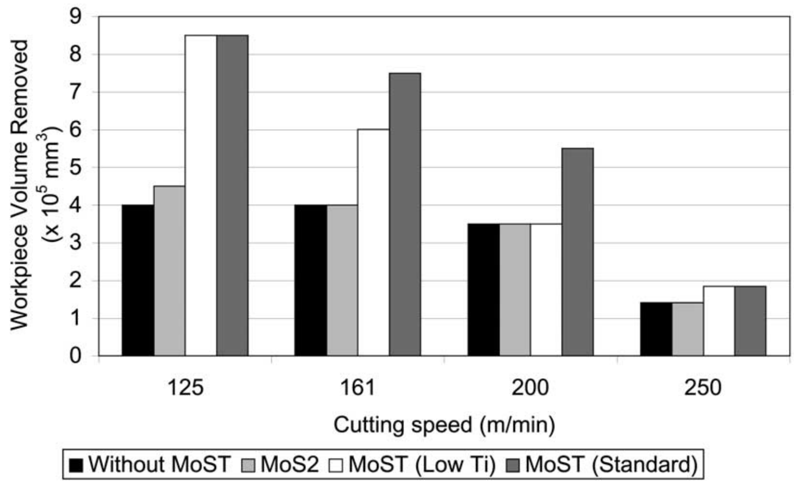

Fig. 13. Dependence of the removed volume of workpiece material up to the end of the tool life of the insert on the cutting speed in milling test. (type 4, 0.25 mm/rev. tooth, aρ = 1.5 mm, d = 80 mm).

Table 4
The summary of the suitability of MoS_2/metal composite coating for milling and turning operations

Machining test	Test condition		Suitability
Milling	Normal	$v_c \leq 250$ m/min $f_z = 0.25$ mm/rev.tooth $a_p = 1.5$ mm	O
	High speed	$v_c > 250$ m/min $f_z = 0.25$ mm/rev.tooth $a_p = 1.5$ mm	X
Turning	$v_c = 150$–250 m/min $f = 0.125$–0.315 mm/rev $a_p = 1.5$–2.5 mm short contact interval operation		O (expected)

O: suitable; X: unsuitable.

properties of the coatings were examined. Ti addition to MoS_2 coating prohibits the micro- or macro-crystalline structure in the coating, and the MoST coating is a single-phase solid solution structure up to 16 at.% Ti. Mechanical properties of the coating are dependent on Ti concentration. The hardness and elastic modulus of the coatings increase with increasing Ti concentration [6]. The residual stresses of the coatings dramatically increased by adding Ti to MoS_2 coating.

Under vacuum, the coatings are macro-crystalline above 500 °C and this results in an increase in coating porosity and a decrease in coating hardness. The concentration of S abruptly decreases for heat treatment temperatures above 750 °C.

In air, the coatings start to oxidise above 400 °C. There are many crystallites in the oxidised surface, which contained a higher Mo fraction and a lower S concentration than the matrix. The concentration of S decreases dramatically if the temperature is above 550 °C. The mechanical properties degrade dramatically by the oxidation.

The wear behaviour of the coatings was examined in turning and milling operations.

In continuous turning experiments: the tool life of the insert coated with MoST is the same as without. Therefore, it is concluded that MoST coating is not suitable for continuous turning operation using type 1 and type 2 inserts. In addition, oxygen was found in the region of the heat-affected zone of the insert after the turning test. This means that oxidation wear of the coating is the dominant wear mechanism in turning operations due to the very high cutting temperature (500–600 °C).

In milling experiments: the tool life of the insert coated with MoS_2/Ti was 1.5–2 times longer than the state-of-the-art tool or the one coated with MoS_2. With very high cutting speed, however, the advantage of MoS_2/Ti coatings is reduced because of the very high cutting temperature. Therefore, the temperature becomes a limiting factor. Consequently, MoS_2/Ti coatings are suitable for dry machining at normal cutting speed.

However, it is suggested from the results of the milling experiments that MoS_2/Ti coating might be suitable for the short contact interval turning operation that is conventionally used in the industry, but this remains for further study. Other geometry and grades could be investigated further.

Acknowledgments

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