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SYNTHESIS OF NEW SUPERHARD MATERIALS AND THEIR APPLICATION TO CUTTING TOOLS

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Summary

Two major areas of effort are encompassed:

I. Synthesis of Superhard Materials: The process of Activated Reactive Evaporation is used to synthesize superhard materials like carbides, oxides, nitrides and ultrafine grain cermets. The deposits are characterized by hardness, microstructure, microprobe analysis for chemistry and lattice parameter measurements. The synthesis and characterization of TiC-Co cermets is given.

II. Machining Evaluation of Coated High Speed Steel Tools. Inserts of Type M-42 High Speed Steel (1/2" x 1/2" x 1/8") were prepared from bar stock and heat treated to a hardness of RC 64 prior to hard coating by the Activated Reactive Evaporation (ARE) process. Screening tests using a fixed set of machining conditions were carried out. The coating variable studies were surface preparation prior to coating, composition of coating (TiC and TiC-10N₄) and biasing of the substrate i.e. 0 volts or ARE process and -2000 Volts or BARE process. In all cases, the cutting forces were markedly lower for the coated tools as compared to the uncoated tools. Examination of the coated tools in the scanning Electron Microscope revealed that the coated tools showed much less wear than the uncoated tools. Some experiments were also tried with TiC coatings on cemented carbide (WC-Co) tool inserts. Again the coated inserts showed much less wear.

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I. OBJECTIVE

The objectives of this research are two-fold: Firstly, the synthesis of new superhard materials. Secondly, to examine the effect of superhard coatings on the performance of high speed steel cutting tools. Such applications as well as others, e.g., grinding tools, hard coatings for abrasion and wear resistance in engines, aircraft, tanks, metal forming machinery, dies, punches, etc., could be of great interest to the DOD.

There are several reasons for concentrating the application effort on cutting tools in general and high speed steel tools in particular. Firstly, very little work has been carried out in the application of hard carbide coatings to improve the life of high speed steel tools (single point tools, end mills, cutters, etc.) even though they form the largest single item in the tool production of cutting tools; the present mill shipments of high speed steel tools is estimated at \$800 million. Secondly, the Activated Reactive Evaporation (ARE) process, recently developed at UCLA under ARPA sponsorship,¹ is uniquely suited for this purpose since the hard coating can be applied even at low substrate temperatures, i.e., about 500°C, which would still retain the substrate (high speed steel tools) in its hardened and tempered condition without softening. Competing processes such as chemical vapor deposition require that the substrate be at temperatures of 1000°C or higher for the deposition of hard compounds such as TiC which would, of course, ruin the strength and toughness of the high speed steel. Such CVD processes are commercially used today to coat TiC onto a WC-Co type carbide machining inserts which can withstand high deposition temperatures (1000°C). Hard coating can also be laid down by sputtering; however, the deposition rates from sputtering are very low.

In the Activated Reactive Evaporation process, Bunshah and his co-workers^{1,2} have developed a new high rate physical vapor deposition process for the synthesis of oxides, nitrides and carbides. Furthermore, they found that by controlling the deposition temperature or by heat-treatment,³ it was possible to change the microstructure and consequently increase the hardness of TiC from 3000 to 5400 kg/mm, a value second only to diamond (see Table I) and comparable to or better than the second hardest material known, borazon (cubic boron nitride). Moreover, TiC should cost 100 times less than borazon or diamond, which is a considerable economic incentive. Finally, high speed steel (a strong and tough material) coated with a superhard layer such as TiC or other materials, would be an ideal system to study for improved tool performance since the necessary requirements of hardness for cutting and toughness to absorb the impact loads are fulfilled by this composite system.

TABLE I
Hardness of Various Materials

Material	Knoop Hardness (kg/mm ²)		Wooddell Scale (relative)
	Room Temperature	900°C	Room Temperature
Diamond	7000		43
Borazon (cubic/boron nitride)	4700		19
Samarium Borides (Sm B _{~2})	3610		
(Sm B _{~70})			
Rare Earth Borides (REB ₇₀)	3500-4000		
Titanium Carbide*	3000	400	
Hafnium Carbide	2400	800	
Vanadium Carbide	2400	400	
Silicon Carbide	2480		14
Niobium Carbide	2200	500	
Aluminum Oxide	2100		9
Tungsten Carbide	1850	1200	
Tantalum Carbide	1700	400	
Chromium Carbide (Cr ₇ C ₃)	1500	600	
Chromium Carbide (Cr ₂₃ C ₆)	950	700	
Quartz	820		7

*The hardness of TiC produced by Activated Reactive Evaporation can be as high as 5400 kg/mm².

II. RESEARCH PROGRAM

The proposed research program is divided into the following two parts:

A. Synthesis of New Superhard Materials

The Activated Reactive Evaporation (ARE) process will be used to synthesize new superhard materials such as carbides, nitrides, oxides and two-phase alloys. The ARE is a versatile process that is capable of the following:

- 1) Synthesizing oxides, carbides, nitrides and possibly borides and silicides.
- 2) Synthesizing mixed carbides (i.e., carbides of more than one metal) of controlled composition, e.g., (TiZr)C.
- 3) Synthesizing mixed compounds (i.e., carbo-nitrides, oxy-carbides, etc.)
- 4) Changing the microstructure and mechanical properties by variation of process parameters, e.g., (deposition temperature).
- 5) Producing material ranging from full density coatings all the way to powder.
- 6) Producing very fine grain sizes.
- 7) Varying and controlling the stoichiometry of carbides and other compounds.
- 8) Laying down a deposit which is strongly adherent to the substrate due to a diffusion bond produced between the two.
- 9) Producing graded microstructures, e.g., from metal to carbide.
- 10) Obtaining high deposition rates for coatings (5 to 25 μ per minute) which make it an economical process.

This part of the research program will be concentrated on the synthesis and characterization of the following superhard materials:

1. Carbides

- a) Carbides of tungsten since they have the highest hot hardness of all carbides.

b) HfC since it has the second highest hot hardness and has been reported to be a good cutting tool coating for machining titanium.

c) Boron carbide since it is a very hard material in current usage.

d) Mixed carbides in the system VC-TiC. Jangg et al⁴ report a maximum in hardness at TiC - 30% VC.

e) Mixed carbides in the system TaC-HfC. The TaC-20% HfC alloy has the highest reported melting point of all alloys.

f) Mixed carbides in the system TiC-ZrC. "Alloys" in this system are reported to be harder than TiC. It is also an experimentally easy alloy to evaporate and will serve as a model system for studying mixed carbides.

2. Nitrides

The synthesis of cubic boron nitride will be attempted since past work has shown that it is possible to synthesize β -SiC⁵ which is also cubic. Success would provide an alternate and possible cheaper method of producing a proven hard material.

3. Oxides

a) Al_2O_3 since this is a commercially used grinding material, and will be used as a model system.

b) Mixed oxides in the system Al_2O_3 -ZrO₂ to produce fine grained two-phase structures of different morphology⁶ harder than the pure oxides.

4. Two-Phase Alloys

a) TiC + B and VC + B - a tenfold increase in strength has been reported due to precipitation hardening.

b) Ultrafine grained cermets-carbides in a matrix of metal or alloy in an effort to improve the toughness of the carbides. The present-day cermets have a lower limit of 3 μ in carbide particle size. The toughness

increases very rapidly as the size of the carbide becomes smaller. Such a very fine grained cermet coating would be expected to retain the cutting ability of the hard carbide while improving its resistance to fracture. Preliminary work at UCLA has shown the ability to synthesize TiC-Ni cermet coating using the ARE process. As a further step, resistance to degradation at high tool tip temperatures would be improved by substituting a more refractory metal for the ductile matrix in the cermet.

c) High temperature ultrafine grained cermets-carbides in a matrix of refractory metal to improve the hot hardness of the composite structure.

The important process variables that will be studied are:

1. Substrate temperature which affects grain size, density, particle size in two-phase alloys and residual stresses. The range of substrate temperatures to be explored is 500-1500°C.

2. Heat treatment - to study annealing effects in single phase material and to study precipitation effects where applicable.

Substrate materials will be a metal and/or ceramic. Deposit thickness will be about .001-.005" so as to assure bulk properties.

Characterization of the synthesized materials will be carried out by the following:

1. Microhardness at room and elevated temperature.
2. Chemical analysis using the microprobe, the non-dispersive detector and other analytical techniques as required.
3. Microstructure using optical, scanning and transmission electron microscopy.
4. X-ray diffraction to measure precision lattice parameters, "particle size" and residual stresses. Computer programs have been developed at UCLA to handle the X-ray data.

5. Density.

At the start of the project, a preliminary study will be run to explore the synthesis of the various materials followed by limited characterization (lattice parameter, microhardness and structure as a function of C/M ratio) to aid material selection for detailed studies.

B. Effect of Superhard Coatings on Cutting Tool Performance

During metal cutting, tool damage can be due to tool wear (flank and crater wear), and/or cracking along the tool face. The tool must support the cutting forces at the high temperatures attained during cutting, and it must also endure the cyclic thermal stresses induced by the cutting, non-cutting cycle. Such conditions call for a tool material that is both hard and tough and resistant to wear.

During this phase of the research program, high speed steel tools will be coated initially with TiC and subsequently with other superhard materials developed in the first part of this program. The coating will provide the necessary hardness and wear resistance while the core, which is the high speed steel tool in this case, will provide the toughness and the resistance to impact loading. Machining tests will be carried out on the coated tools at different conditions of cutting speeds, feed, and depth of cut. Intermittant as well as non-intermittant tests will be employed. The cutting forces will be measured in each case using a tool dynamometer. The tool work interface temperature and friction coefficient along the tool face will be determined. Tool damage and surface finish of the machined part will be examined. Tool life will be determined. The wear process and mechanism of the different coatings will be investigated using tool-maker's microscope, scanning electron microscope techniques, and electron microprobe analyses. The crater and flank wear heights will be measured at different locations. The scanning electron

microscope brings out topographical details of tool wear, cracks, and failure with more clarity and detail than one can observe with an optical microscope. While a SEM photograph provides fine details of tool surfaces, it is still necessary to have the electron microprobe analysis for identification of specific elements in the worn tool surfaces which then helps to understand the contact interactions between workpiece and tool. Correlation between microstructure and mechanical properties of the coating and the core material to their cutting performance will also be examined. Comparison of the cutting forces, temperature, friction coefficient, surface finish of the machined part, and tool wear will be examined and correlation to the physical and mechanical properties of the different coatings will be carried out.

Tool damage vs. cutting speed for a given feed and depth of cut will be compared for different tool coatings. Wear measurements will be obtained from direct comparison of the tool contours before and after test. Correlation of tool wear results to parameters characterizing the coating (coating composition, grain size, density, lattice parameter, coating thickness, microhardness and C/M ratio) will be investigated. Such correlation will constitute the feedback information for the synthesis of the superhard coating materials.

III. PROGRESS TO DATE

In this reporting period, work was concentrated into the areas as reported below:

Synthesis of TiC-Co Cermets

a) Objective To synthesize TiC-Co cermets by the Activated Reactive Evaporation (ARE) process and characterize them by microhardness, lattice parameter and chemical composition using the electron microprobe analyzer.

b) Experimental Procedure In the synthesis of TiC-Co cermets by the ARE process, an alloy rod of Ti-Co is evaporated in the presence of the reactive gas C_2H_2 . Titanium reacts with the gas and forms TiC. Co remains as metal because the free-energy of formation for Co carbides is not favorable for the reaction to take place ($\Delta G_{298}^{\circ} = + 6.9K \text{ cal/mol}$ for Co_3C).

Two experiments were made to synthesize TiC-Co cermets at the conditions given in Table I. In run A-1, Ti-Co alloy was deposited first in the absence of C_2H_2 and then the reactive gas was introduced to form TiC-Co cermet. In the run A-2, the shutter was opened after introducing C_2H_2 to deposit only TiC-Co. Lattice parameters of TiC in the deposits were determined using a Norelco x-ray diffractometer. A Micromet microhardness tester was used to measure knoop hardnesses of the deposits with 50 g. load. The results are also shown in Table I.

To determine the chemical composition of the deposits across the cross-section, a sample from deposit A-1 was electroplated with copper and was mounted in cross-section along with a piece of Ti-Co evaporant rod as a standard. The mount was given a metallographic polish and the deposit and the standard were analyzed by electron microprobe analysis on an energy dispersive analyzer (Kevex unit) attached to a scanning electron microscope.

TABLE I

Synthesis of Ti-Co Cermets

Run #	Evaporation rate g/min.	Deposition rate mil/min.	Reactive gas pressure torr	Temp. of deposition °C	Hardness KHN kg/mm ²	Lattice parameter A°
Ti-Co-A-1	0.17	0.032	$4-6 \times 10^{-4}$	700	2095	4.320 ± 0.002
Ti-Co-A-2	0.18	0.036	$6-8 \times 10^{-4}$	700	2030	$4.320 \pm .001$

The electron beam was moved across the thickness of the deposit. When the beam was moving, the intensity of x-rays from each element was traced. Figure 1 shows the microprobe traces relative to the amounts of Ti and Co present in the standard specimen and across the cross-section of the deposit. An electron micrograph of the deposit showing Ti-Co alloy and TiC-Co cermet layers is shown in Figure 2.

c) Results and Discussion The variation in chemical composition of the deposit A-1 can be discussed with the help of Figs. 1 and 2. The first layer on the stainless steel substrate is the Ti-Co alloy layer (see fig. 2). The variation in composition of Ti and Co are within the scatter band for these elements for the standard specimen (see fig. 1).

There is a drop in Ti concentration at the boundary between the Ti-Co and the next layer TiC-Co.

In the standard, Ti/Co x-ray intensity ratio is 1.53 and corresponds to 16.2 atomic ratio. X-ray intensity ratios of Ti/Co in the TiC-Co cermet region were calculated at points A, B and C as marked in Fig. 1. The intensity ratios varied from 1.35 to 1.59 corresponding to atomic ratios of 14.3 to 16.9. Thus the Ti/Co ratio in the TiC-Co cermet is very close to that in the Ti-Co standard within experimental error and the intensity limit bars of the elements in the standard as shown in Fig. 1.

The drop of x-ray intensities of Ti and Co in the last 5.34 μ m region corresponds to the step formed due to the polishing off of the soft copper coating as can be seen in Fig. 2.

Lattice parameters of TiC in the deposits are 4.320 and correspond to a composition of TiC. 65. The hardnesses of the deposits are comparable to WC-Co cermets made by powder metallurgy techniques (\sim 2000 KHN).

d) Conclusions X-ray diffraction of the deposits shows the presence of $TiC_{.65}$ in the cermets. The Ti/Co ratio in the deposits is shown to be approximately the same as that in the evaporant alloy rod by electron microprobe analysis. The microhardnesses of the deposits are comparable to WC-Co cermets made by powder metallurgy techniques. These results show that TiC-Co cermets can be synthesized by activated reactive evaporation from a single rod-fed alloy source. Further experiments will be carried out changing the experimental parameters such as deposition temperature, alloy evaporation rate and reactive gas pressure to synthesize and characterize the TiC-Co cermets.

2. Machining Evaluation of Coated High Speed Steel

In the last semi-annual report for the period October 1975-March 1976 (UCLA-ENG-7659), it was reported that the high speed steel inserts (Type M43) purchased from a commercial producer were unsatisfactory for coating because of the rough grinding and turned-up edges. Therefore, we produced our own high speed steel inserts from Type M42 high speed steel by purchasing a rod of the material, machining out inserts (1/2" x 1/2" x 1/8" thick) and heat treating them to a hardness of RC 64. All work was done on these inserts.

The screening tests were carried out under the following conditions:

Speed = 150 rpm

Feed = .003 in/rev

Depth of cut = .040 inches

Rake angle $\alpha = +5^\circ$

The machining time was kept constant at 2 minutes. The workpiece was a bar of Type 4340 steel heat treated to a hardness of RC 22.

In order for a hard coating to be effective in prolonging tool life, it must adhere to the substrate under the loads imposed in the cutting test. A very favorable indication of a good coating is the reduction in the tool cutting forces as compared to an uncoated tool. This will lead to reduced tool tip temperature and rate of tool wear. The screening test was designed to evaluate these factors. A further indication of tool wear is obtained from photomicrographs of the wear surface after testing for which the scanning Electron Microscope is ideal because of its good depth of field.

The Adhesion of a coating to a substrate consists of two components - mechanical adhesion by locking of the coating with the asperities on the surface of the tool and a diffusion bond between the two. The latter is promoted by temperature and time. A farther obvious consideration is the removal of contaminants like oil or grinding compound which would cause difficulties with the formation of a diffusion bond.

One way to improve the diffusion bonding is to increase the temperature locally at the deposition interface. This can be conveniently done by biasing the substrate to a negative potential which increases the kinetic energy of the fraction of the flux of atoms which have been ionized in the plasma of the ARE process. This results in a higher localized temperature at the coating interface and should increase the depth of the diffusion zone between the substrate and the coating. This was studied for a number of coatings TiC, TiC-Co, ZrC, TaC, NbC. In all cases, the diffusion zone width was $3\mu\text{m}$ approximately in the coatings produced with a substrate bias as compared to $1\mu\text{m}$ with an unbiased substrate.

This is called the BARE process and was originally reported in the Semi-Annual Progress Report for the period October 1975-March 1976
UCLA-ENG-7659.

The coating variables studied were surface preparation prior to coating composition of the coating (TiC and TiC-10N₁) biasing of the substrate i.e. ARE or BARE process, and whether the inserts were rotated during coating or the coating was applied in sequential steps firstly to the flank faces and then to the rake face in an orthogonal coating direction. The direction of coating has been found to be important, the orthogonal coating flux impingement producing the hardest coatings. Thus coatings on rotated inserts are expected to be softer than those of sequentially coated inserts. The results are shown in Table III.

The results show that for all conditions of surface preparation, ARE or BARE process, rotation during coating or sequential deposition, the cutting forces were considerably smaller than those for the uncoated inserts.

Fig. 3 a and 3b show the rake face and flank face of the uncoated inserts. Notice the extensive wear on the nose of the tool and on the rake face where the edge has been worn away. By contrast figs. 4a, 4b, 5, 6, 7 show inserts nos. 47, 88, 87 and 81 showing much smaller wear on both the rake and flank faces. Insert 47 is outstanding showing almost zero wear even though the coating had chipped

on the rake face prior to machining due to feathered edge on the uncoated insert. Note that the coating does not come up to the edge even outside the machined zone. Fig. 8 shows insert No. 83 with considerable wear of the coating but hardly any wear of the insert. Energy dispersive x-ray analysis of points 1,2, and 3 show the presence of Ti at all the locations thus indicating that the coating is still present but shows some wear. Thus the formation of the diffusion zone between the coating and substrate is very beneficial in prolonging tool life.

The uncoated insert also exhibits a greater fluctuation in cutting forces.

The next step in evaluating the various coatings is to run tool life tests which will be accomplished in the next reporting period.

3. Machining Evaluation of Coated Carbide Inserts

Coated carbide inserts by the CVD process are widely used in industry. Recently Nakamura et.al.⁽⁷⁾ published a paper showing that carbide inserts coated with TiC using the ARE process had less crater and flank wear than the CVD method TiC coated inserts and equal to the CVD method TiC-Ti(C+N)-TiN coated inserts. Therefore, a few tests were carried out by coating Grade 6 carbide inserts with TiC using the ARE process. They were subject to the same type of screening tests as the coated high speed steel inserts. The results are shown in Table III. There is not such a marked decrease in tool cutting forces as with high speed steel inserts. This is to be expected since in one case a comparison is being made between a high speed steel surface and a carbide surface which are two different materials and in the other case between two fairly similar materials i.e. WC-Co and TiC. Figs. 9a, 9b, 10a and 10b show the wear pattern on uncoated and coated tools and it is clearly seen that the coated tools have markedly reduced wear on both the rake face and flank face.

It may be concluded that hard overlay coatings produce marked improvement in decreasing the wear rate of high speed steel and carbide tools.

IV. FUTURE RESEARCH EFFORTS

Research effort in the next period will consist of:

1. Synthesis and evaluation of TiC-B, TaC-HfC coatings
2. Evaluation of the tool life of coated vs. uncoated high speed steel inserts.

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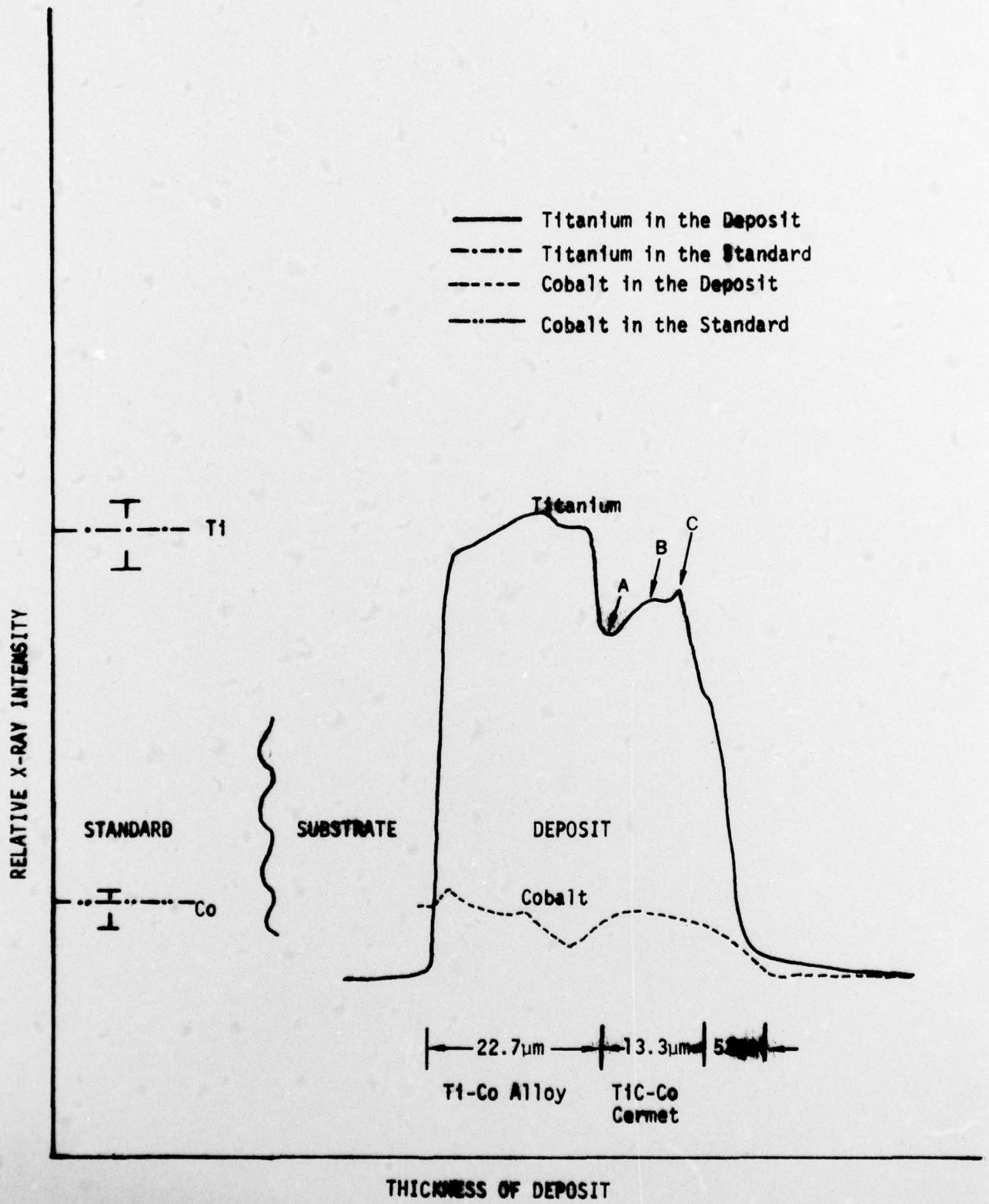


Figure 1. Compositional Variations of the Elements Ti and Co across the Thickness of TiC-Co Deposit A-1 and the Standard Ti-Co Evaporant Rod.

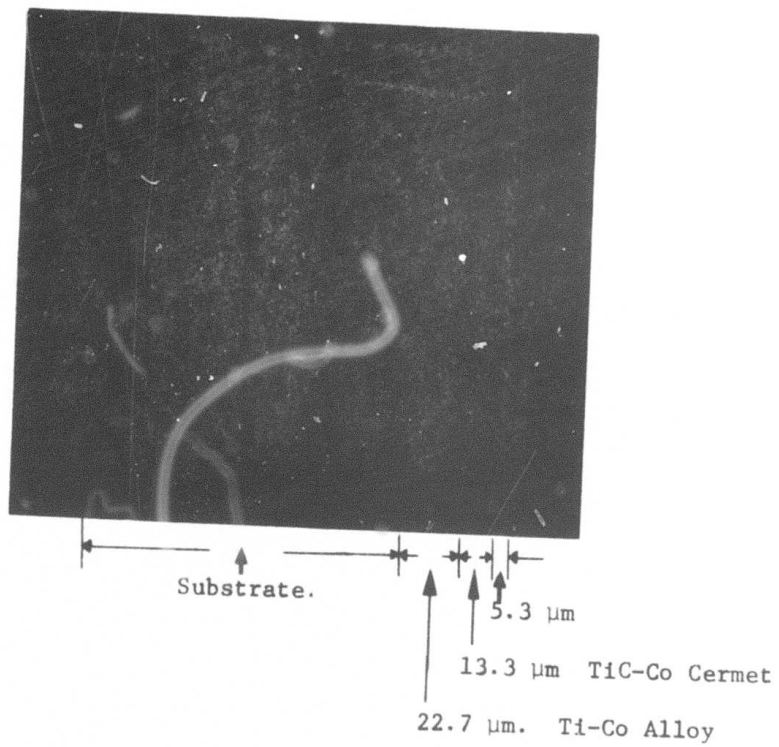
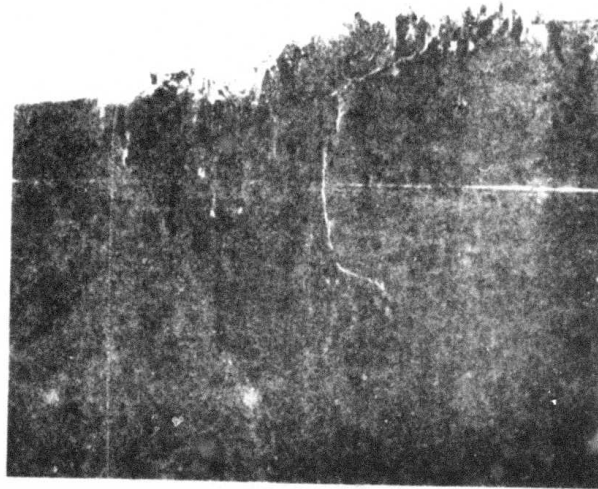
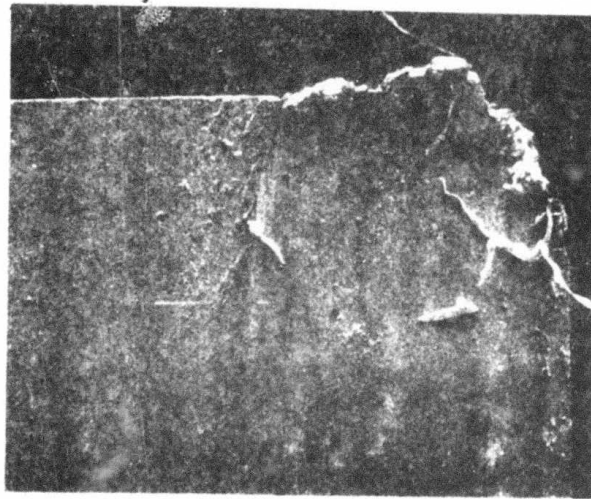


Figure 2. Electron Micrograph of Deposit A-1 in Cross-Section Showing Ti-Co Alloy and TiC-Co Cermet Layers. 375X

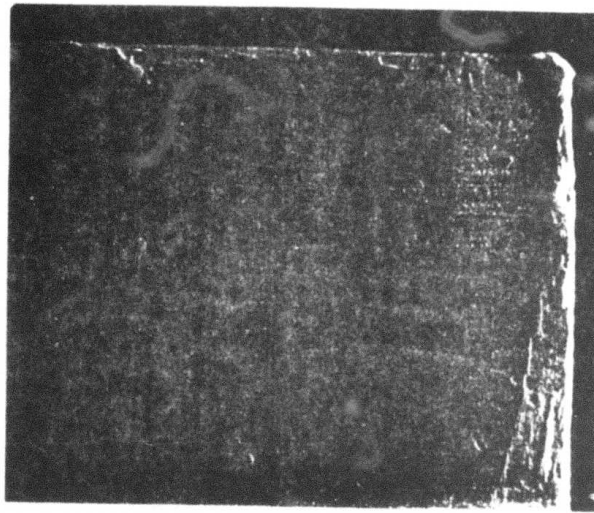


(a)

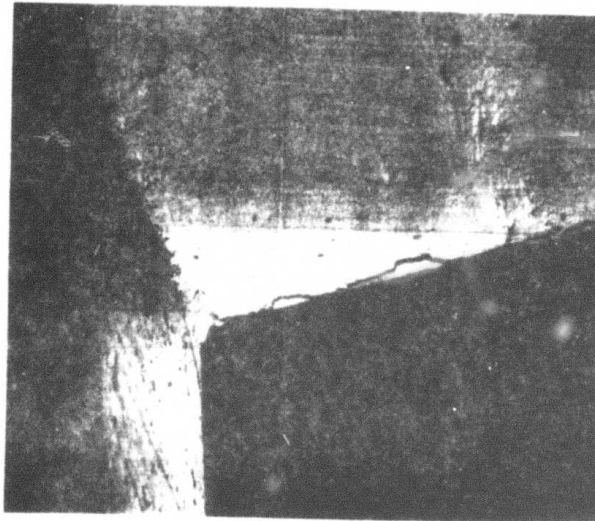


(b)

Fig. 3 - Photomicrographs of an uncoated M-42 high speed steel tool after machining.



(a)



(b)

Fig. 4 - Rake face and isometric view of coated tool 47 after machining showing negligible tool wear.

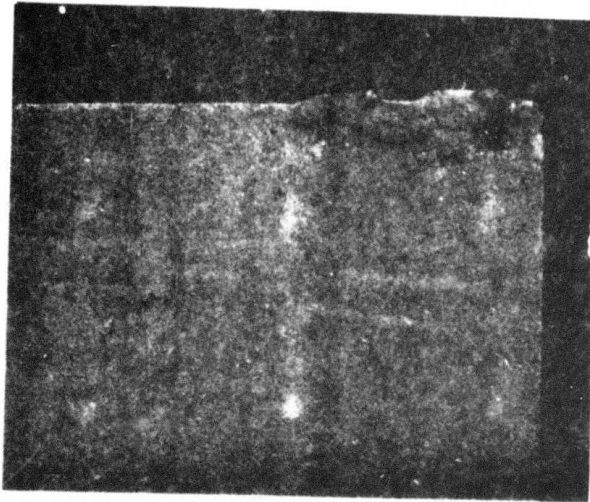


Fig. 5 - Photomicrograph of rake
face of coated tool 88 after
machining.

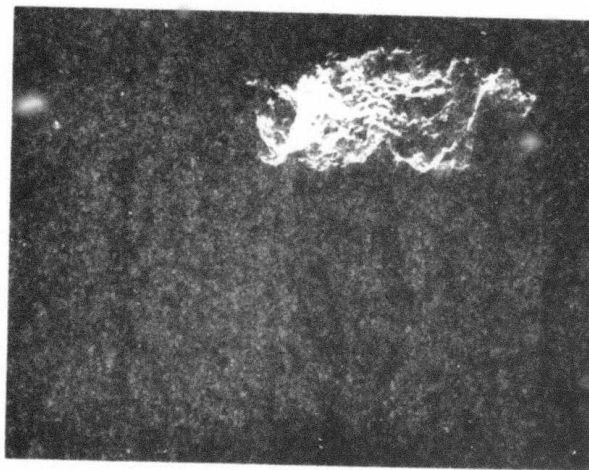


Fig. 6 - Photomicrograph of rake
face of coated tool 81 after
machining.



Fig. 7 - Isometric view of coated tool 87 after machining.

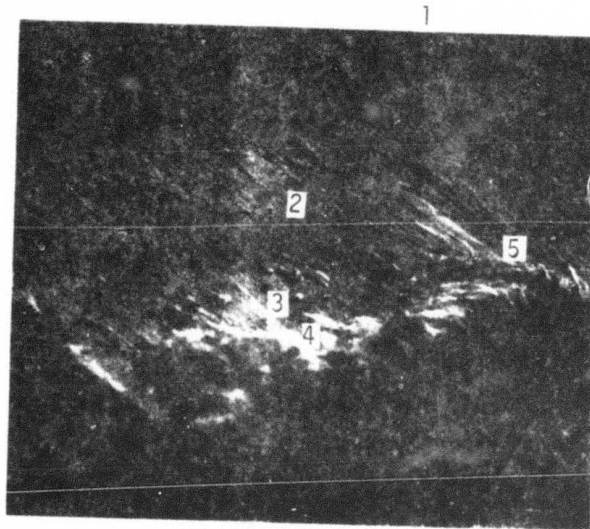
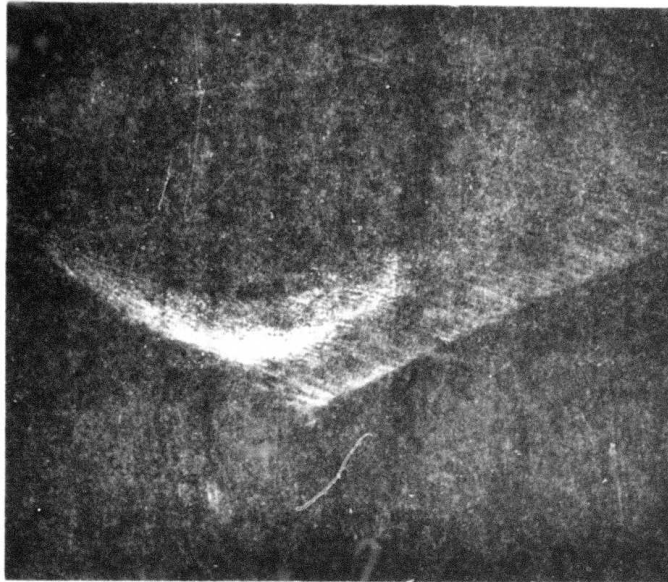
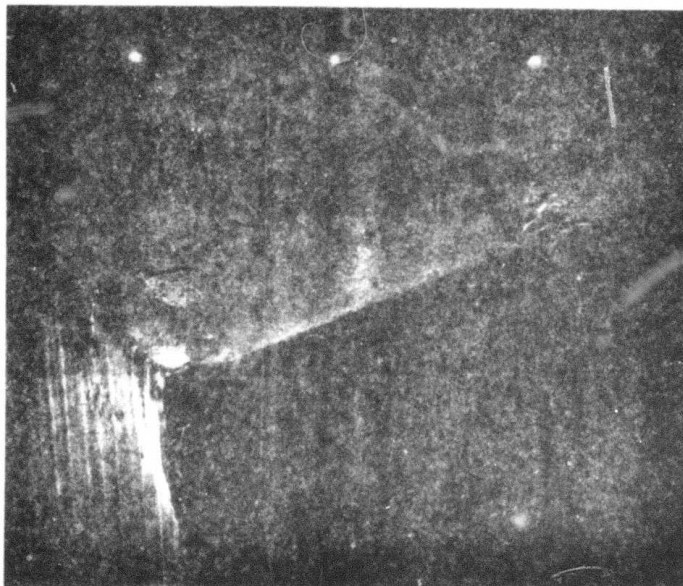


Fig. 8 - Isometric view of coated tool 83 after machining.

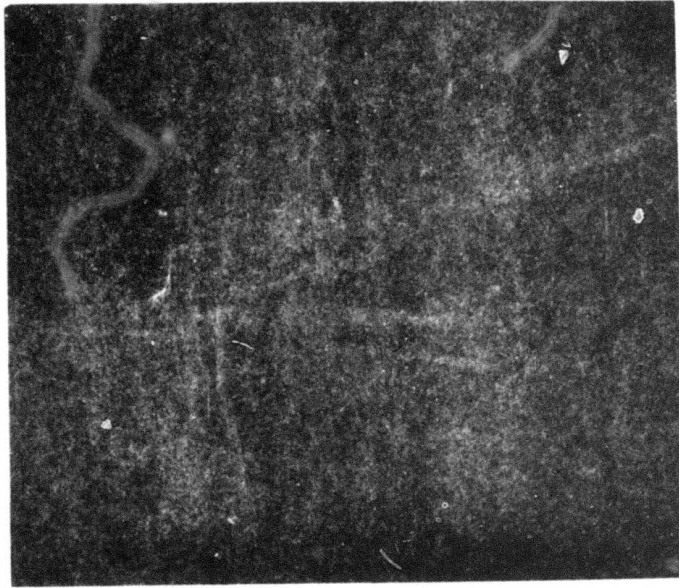


(a) Uncoated carbide

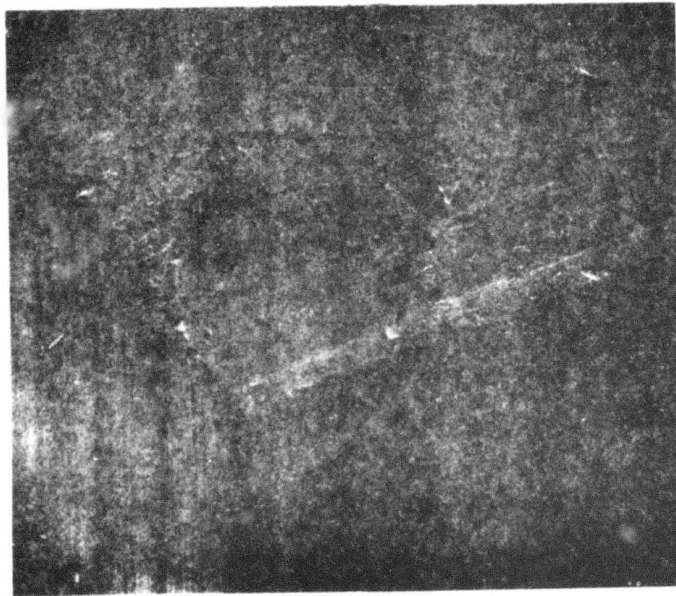


(b) Coated carbide (less flank wear)

Fig. 9 - Isometric view of carbide tools after machining.



(a) Uncoated carbide tool



(b) Coated carbide tool (wear is reduced)

Fig. 10 - Isometric view of carbide tools after machining.

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ACTIVATED REACTIVE EVAPORATION DEPOSITION CARBIDES NITRIDES OXIDES	CERMETS TITANIUM CARBIDE SYNTHESIS OF MATERIALS CUTTING TOOLS PERFORMANCE	MACHINING COATINGS COATED CUTTING TOOLS TOOL LIFE
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<p>The process of Activated Reactive Evaporation (ARE) is used to synthesize superhard materials like carbides, oxides, nitrides and ultrafine grain cermets. The deposits are characterized by hardness, microstructure, microprobe analyses for chemistry and lattice parameter measurements. The synthesis and characterization of TiC-Co cermets is given. Machining evaluation of coated high speed tool steel inserts of type M-42 high speed steel (1/2" X 1/2" X 1/8") were prepared from bar stock and heat treated to a hardness of RC 64 prior to hard coating by the ARE process. Screening tests using a fixed set of machining conditions were</p>		

carried out. The coating variables studied were surface preparation prior to coating, composition of the coating (TiC and TiC-10Ni) and biasing of the substrate; i.e., 0 volts or ARE process and -2000 volts or biased ARE process. In all cases, the cutting forces were markedly lower for the hard coated tools as compared to the uncoated tools. Examination of the coated samples in the scanning electron microscope revealed that the coated tools showed much less wear than the uncoated tools. Some experiments were also tried with TiC coating on cemented carbide (WC-Co) tool inserts. Again, the coated inserts showed much less wear so it is to be concluded that the effect is consistent; hard coatings of the type investigated do exhibit improved machinability.

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