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Effects of Composition and Processing Variables on Transverse Rupture Strength and Hardness of Nickel-Alloy-Bonded Titanium Carbide

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UNITED STATES DEPARTMENT OF THE INTERIOR

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

Å	angstrom	lb	pound
atm	atmosphere	min	minute
°C	degree Celsius	mL	milliliter
°C/min	degree Celsius per minute	mm	millimeter
cm	centimeter	μm	micrometer
g	gram	pct	percent
g/cm^3	gram per cubic centimeter	psi	pound per square inch
h	hour	rpm	revolution per minute
HRA	Rockwell A hardness	st	short ton
ksi	kips per square inch	st/in^2	short ton per square inch
L	liter	wt pct	weight percent
L/h	liter per hour		

EFFECTS OF COMPOSITION AND PROCESSING VARIABLES ON TRANSVERSE RUPTURE STRENGTH AND HARDNESS OF NICKEL-ALLOY-BONDED TITANIUM CARBIDE

By G. T. Fisher II,[1] L. L. Oden,[2] and G. Asai[1]

ABSTRACT

The material requirements of carbide cutting tools for machining steel and cast iron are dependent on WC bonded with Co, with additions of TaC and TiC. The Bureau of Mines is conducting research to devise substitute materials, based on Ni-alloy-bonded TiC, for C-5-grade cutting tool applications. This substitution would result in less dependence on foreign sources for Co, Ta, and W, and use of lower cost materials. In this preliminary study, the Bureau investigated the effects of composition and processing variables on the transverse rupture strength and hardness of Ni-alloy-bonded TiC to identify compositions that exhibit promising mechanical properties. Further systematic modification of these compositions by soild-solution strengthening additions would subsequently be evaluated to obtain cutting tool materials with requisite properties and performance for C-5 applications. Compositions for evaluation were prepared by a powder metallurgy method of cold-pressing and liquid-phase sintering. Complementary analysis of compositions included chemical analysis, X-ray diffraction, energy-dispersive X-ray spectroscopy, and density measurements.

Based on results of this investigation, promising compositions were determined to be 59.37TiC-25Ni-5Mo-10.63Mo_2C, 59.68TiC-25Ni-3.33Mo-1.77Mo_2C-6.67W-3.55WC, and 59.35TiC-25Ni-1.67Mo-3.54Mo_2C-3.33W-7.1WC.

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INTRODUCTION

The material requirements of cemented carbide tools for machining cast iron and steel are based on Co-bonded WC (tungsten carbide), with additions of TaC (tantalum carbide) and TiC (titanium carbide). In 1984, U.S. consumption of these materials for cutting and wear-resistant applications comprised 14.1 million lb W as WC metal powder (1),[3] 831,000 lb of contained Co (2), and 129,000 lb of contained Ta (3). Cemented carbide cutting tools used in C-5[4] applications consume a substantial portion of these strategic and critical materials. In 1983, approximately 40 pct of all cemented carbide production was used for cutting tools (4). About 35 pct of all machining is done using C-5 grade tools (5). Commercial tools for C-5 applications contain approximately 71WC-11.5TaC-8TiC-9.5Co.[5] No commercial reserves of Co and Ta are located in the United States.

To address this problem, the Bureau of Mines is conducting research to devise substitutes for the strategic and critical materials in C-5-grade cutting tools. The principal characteristics of a cutting tool are its toughness and wear resistance, indicated by TRS (transverse rupture strength) and hardness, respectively. In this preliminary study, the effects of composition and processing variables on the TRS and hardness of Ni-alloy-bonded TiC are described. Compared to WC, TiC was selected because of its superior properties, which include high hardness, high oxidation resistance, low density, and low thermal conductivity. The objective of this study was to delineate promising compositions with best combination of TRS and hardness. Further systematic modification of these compositions by solid-solution strengthening additions would subsequently be evaluated to devise cutting tool materials with the requisite mechanical properties and machinability performance for C-5 applications. Successful prosecution of this research would result in less dependence on foreign sources for Co, W, and Ta, and use of lower cost domestic materials.

TiC-based hard metals have been commercially available since the early 1930's but have had few applications owing to their brittleness (6). At the end of the 1950's, TiC with a Ni-Mo binder became useful for finish machining of steel (7), where high toughness was not required. Problems with these early bonded TiC hard metals were associated with grain growth on sintering, resulting in low strength. Humenik (8) related grain growth in Ni-bonded TiC to incomplete wetting and demonstrated that additions of Mo, upon sintering, resulted in a mixed carbide phase, (Ti, Mo)C, that exhibited good wetting compared to TiC and retarded grain growth. Thus, hardness and impact resistance of these materials were increased. Other investigators report similar findings on the formation of the mixed carbide phase and its beneficial effects (9-12). Moskowitz (13-14) reported that a TiC-based C-5-grade tool for rough machining of steel contained 66.9TiC-22.5Ni-10.62Mo_2C and exhibited TRS and hardness values of 275,000 psi and 90.6 HRA, respectively. A comprehensive compilation of commercially available WC- and TiC-based cutting tools was presented by Brooks (15). Notwithstanding the historical development of TiC-based tools, a satisfactory substitute for WC-Co tools for C-5 applications has not been developed owing to inadequate toughness of the bonded TiC tools.

[3]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

[4]The C-5 category of cutting tools are used for milling and rough turning of most steels--hardened, alloy, and mild--and also for certain Ni- and Fe-base high-temperature alloys and for cast iron.

[5]Compositions in this report are expressed in weight percent.

EXPERIMENTAL PROCEDURES

EXPERIMENTAL DESIGN

Two factorial experiments were designed to investigate single-element and interaction effects of composition on the TRS and hardness of Ni-Mo-Mo_2C- and Ni-Mo-Mo_2C-W-WC-bonded TiC. Calculated compositions and variables are summarized in tables 1 and 2. For the first system, the following factorial experimental design was selected:

1. TiC at three levels (60, 70, and 80 wt pct).
2. Mo:(Mo+Ni) ratio varied at four levels (1/8, 3/8, 5/8, and 7/8).
3. C at four levels equivalent to 0, 1/3, 2/3, and 3/3 of the Mo expressed as Mo_2C.

To verify the reproducibility of the data, subsequent sintering runs were done on a quarter-replicate of the alloys tested. The following experimental design was selected:

1. Three levels of TiC (60, 70, and 80 wt pct).
2. For each TiC level, the Mo:(Mo+Ni) ratio was varied at 1/8, 3/8, 5/8, and 7/8; the corresponding fraction of Mo added as Mo_2C was 1/3, 2/3, 0, and 1, respectively.

For the second system, the following factorial experimental design was selected:

1. TiC at three levels (60, 70, and 80 wt pct).
2. Constant Ni:(Mo+W) ratio of 5/3.
3. W substituted for Mo at four levels (0, 1/3, 2/3, and 3/3).
4. C at four levels equivalent to 0, 1/3, 2/3, and 3/3 of the Mo and W added as Mo_2C or WC.

A quarter-replicate of the alloys tested were sintered in subsequent runs. The following design was selected:

1. Three levels of TiC (60, 70, and 80 wt pct).
2. Constant Ni:(W+Mo) ratios of 5/3.
3. For W:(W+Mo) ratios of 0, 1/3, 2/3, and 3/3, the corresponding fraction of Mo and W added as carbides was 2/3, 0, 1/3, and 3/3, respectively.

To investigate the effect of sintering time at 1,400° C on the TRS and hardness of Ni-alloy-bonded TiC, two series of four compositions based on 60TiC-25Ni were selected, with W substituted for Mo at four levels--0, 1/3, 2/3, and 3/3. For the first series, Mo and W were added as elemental constituents. For the second series, Mo and W were added as Mo_2C or WC. Sintering time was investigated over the range of 0.5 to 240 min. Lattice parameters of carbide and binder phases were determined by X-ray diffraction. Scanning electron microscopy using energy-dispersive X-ray spectroscopy (EDAX) was used to determine the relative amounts of elements in the TiC cores, solid-solution layer (rim) around the TiC cores, and Ni-alloy binder phase. The effect of sintering temperature and soak time at final sintering temperature on the TRS of alloy 58 was studied. Final sintering temperatures of 1,400°, 1,500°, and 1,600° C and soak times of 30, 60, and 120 min were investigated.

MILLING AND BLENDING OF POWDERS

Compositions were prepared using high-purity powders with a particle size range of 1 to 5 μm. Table 3 lists the constituent powders used, suppliers,[6] and analyzed carbon and oxygen contents. Batches with a total powder weight of 400 g were prepared using the procedure described herein. Constituent powders were weighed on a balance in an He-filled glovebox in the absence of air and were

[6]Reference to specific manufacturers does not imply endorsement by the Bureau of Mines.

TABLE 1. - Composition and variables for factorial experimental design of Ni-Mo-alloy-bonded TiC

Alloy	Composition, wt pct				Variables		Alloy	Composition, wt pct				Variables	
	TiC	Ni	Mo	Mo_2C	Mo:(Mo+Ni)	Fraction of Mo as Mo_2C, pct		TiC	Ni	Mo	Mo_2C	Mo:(Mo+Ni)	Fraction of Mo as Mo_2C, pct
1.....	60.00	35.00	5.00	0	0.125	0	25....	70.00	11.25	18.75	0	0.625	0
2.....	59.90	35.00	3.33	1.77	.125	33	26....	69.61	11.25	12.50	6.64	.625	33
3.....	59.79	35.00	1.67	3.54	.125	67	27....	69.22	11.25	6.25	13.28	.625	67
4.....	59.69	35.00	0	5.31	.125	100	28....	68.83	11.25	0	19.92	.625	100
5.....	60.00	25.00	15.00	0	.375	0	29....	70.00	3.75	26.25	0	.875	0
6.....	59.69	25.00	10.00	5.31	.375	33	30....	69.45	3.75	17.50	9.30	.875	33
7.....	59.37	25.00	5.00	10.63	.375	67	31....	68.90	3.75	8.75	18.60	.875	67
8.....	59.06	25.00	0	15.94	.375	100	32....	68.36	3.75	0	27.89	.875	100
9.....	60.00	15.00	25.00	0	.625	0	33....	80.00	17.50	2.50	0	.125	0
10....	59.48	15.00	16.67	8.85	.625	33	34....	79.94	17.50	1.67	.89	.125	33
11....	58.96	15.00	8.33	17.71	.625	67	35....	79.90	17.50	.83	1.77	.125	67
12....	58.44	15.00	0	26.56	.625	100	36....	79.84	17.50	0	2.66	.125	100
13....	60.00	5.00	35.00	0	.875	0	37....	80.00	12.50	7.50	0	.375	0
14....	59.27	5.00	23.33	12.40	.875	33	38....	79.84	12.50	5.00	2.66	.375	33
15....	58.54	5.00	11.67	24.79	.875	67	39....	79.69	12.50	2.50	5.31	.375	67
16....	57.81	5.00	0	37.19	.875	100	40....	79.53	12.50	0	7.97	.375	100
17....	70.00	21.25	8.75	0	.292	0	41....	80.00	7.50	12.50	0	.625	0
18....	69.82	21.25	5.83	3.10	.292	33	42....	79.74	7.50	8.33	4.43	.625	33
19....	69.63	21.25	2.92	6.20	.292	67	43....	79.48	7.50	4.17	8.85	.625	67
20....	69.45	21.25	0	9.30	.292	100	44....	79.22	7.50	0	13.28	.625	100
21....	70.00	18.75	11.25	0	.375	0	45....	80.00	2.50	17.50	0	.875	0
22....	69.77	18.75	7.50	3.98	.375	33	46....	79.63	2.50	11.67	6.20	.875	33
23....	69.53	18.75	3.75	7.97	.375	67	47....	79.27	2.50	5.83	12.40	.875	67
24....	69.30	18.75	0	11.95	.375	100	48....	78.90	2.50	0	18.60	.875	100

TABLE 2. - Composition and variables for factorial experimental design of Ni-Mo-W-alloy-bonded TiC

Alloy	Composition, wt pct						Variables	
	TiC	Ni	Mo	Mo_2C	W	WC	W:(W+Mo)	Fraction of W and Mo added as Mo_2C or WC, pct
49.....	60.00	25.00	15.00	0	0	0	0	0
50.....	59.69	25.00	10.00	5.31	0	0	0	33
51.....	59.37	25.00	5.00	10.63	0	0	0	67
52.....	59.06	25.00	0	15.94	0	0	0	100
53.....	60.00	25.00	10.00	0	5.00	0	.33	0
54.....	59.69	25.00	6.67	3.54	3.33	1.77	.33	33
55.....	59.39	25.00	3.33	7.08	1.67	3.55	.33	67
56.....	59.05	25.00	0	10.62	0	5.33	.33	100
57.....	60.00	25.00	5.00	0	10.00	0	.67	0
58.....	59.68	25.00	3.33	1.77	6.67	3.55	.67	33
59.....	59.35	25.00	1.67	3.54	3.33	7.10	.67	67
60.....	59.04	25.00	0	5.31	0	10.65	.67	100
61.....	60.00	25.00	0	0	15.00	0	1.00	0
62.....	59.67	25.00	0	0	10.00	5.33	1.00	33
63.....	59.35	25.00	0	0	5.00	10.65	1.00	67
64.....	59.02	25.00	0	0	0	15.98	1.00	100
65.....	70.00	18.75	11.25	0	0	0	0	0
66.....	69.77	18.75	7.50	3.98	0	0	0	33
67.....	69.53	18.75	3.75	7.97	0	0	0	67
68.....	69.30	18.75	0	11.95	0	0	0	100
69.....	70.00	18.75	7.50	0	3.75	0	.33	0
70.....	69.76	18.75	5.00	2.66	2.50	1.33	.33	33
71.....	69.53	18.75	2.50	5.31	1.25	2.66	.33	67
72.....	69.29	18.75	0	7.97	0	3.99	.33	100
73.....	70.00	18.75	3.75	0	7.50	0	.67	0
74.....	69.76	18.75	2.50	1.33	5.00	2.66	.67	33
75.....	69.51	18.75	1.25	2.66	2.50	5.33	.67	67
76.....	69.28	18.75	0	3.98	0	7.99	.67	100
77.....	70.00	18.75	0	0	11.25	0	1.00	0
78.....	69.76	18.75	0	0	7.50	3.99	1.00	33
79.....	69.51	18.75	0	0	3.75	7.99	1.00	67
80.....	69.27	18.75	0	0	0	11.98	1.00	100
81.....	80.00	12.50	7.50	0	0	0	0	0
82.....	79.84	12.50	5.00	2.66	0	0	0	33
83.....	79.69	12.50	2.50	5.31	0	0	0	67
84.....	79.53	12.50	0	7.97	0	0	0	100
85.....	80.00	12.50	5.00	0	2.50	0	.33	0
86.....	79.85	12.50	3.33	1.77	1.67	.88	.33	33
87.....	79.68	12.50	1.67	3.54	.83	1.78	.33	67
88.....	79.53	12.50	0	5.31	0	2.66	.33	100
89.....	80.00	12.50	2.50	0	5	0	.67	0
90.....	79.84	12.50	1.67	.88	3.33	1.78	.67	33
91.....	79.68	12.50	.83	1.77	1.67	3.55	.67	67
92.....	79.51	12.50	0	2.66	0	5.33	.67	100
93.....	80.00	12.50	0	0	7.50	0	1.00	0
94.....	79.84	12.50	0	0	5.00	2.66	1.00	33
95.....	79.67	12.50	0	0	2.50	5.33	1.00	67
96.....	79.51	12.50	0	0	0	7.99	1.00	100

TABLE 3. - Stock powders used to prepare experimental compositions

Powder	Supplier	Analysis, wt pct	
		C	O
TiC	H. C. Starck, Berlin, West Germany	19.2	0.26
Ni	Alcan Ingot and Powders, Union, NJ	.07	.07
Mo	Amax Specialty Metals Corp., Coldwater, MI	.01	.16
Mo_2C	Atlantic Equipment Engineers, Bergenfield, NJ	5.90	.18
W	Consolidated Astronautics, Hauppague, NY	.01	.34
WC	Teledyne Wah Chang, Huntsville, AL	6.15	.07

placed in a 11.43-cm-diam by 16.5-cm-long Ti mill. A pressing aid of Carbowax 600, constituting 4 wt pct of total powder charge, was added. The wax also acts to coat the surfaces of the milled powders and protects them from oxidation during handling and cold pressing of the powder. A milling medium of 2,400 g of 4.8-mm-diam TiC-Ni-Mo balls was added. For preliminary tests, a milling solvent of 600 mL hexane was added. Subsequent tests on quarter-replicate alloys used 235 mL acetone as the milling solvent to improve wax distribution in the pressed compacts. Mills were filled with Ar prior to milling. The charge was subsequently milled for 100 h at 75 rpm.

SCREENING, DRYING, AND STORAGE OF MILLED POWDERS

The milled powder slurry and carbide balls were separated using a 4-mesh sieve. The milling solvent was allowed to evaporate in air until the powder was dry enough to be screened using a 30-mesh sieve. The powder was subsequently dried in a vacuum oven at 50° C for 8 h to remove any residual milling solvent and moisture. Dried powder compositions were stored in mason jars filled with Ar to prevent exposure to air and moisture.

PRESSING OF EXPERIMENTAL COMPOSITIONS

Powder compositions were pressed into rectangular shapes using the 50-st hydraulic press and hardened steel die assembly shown in figure 1. Powder compacts for TRS and hardness tests prior to the sintering operation were 0.3 by 0.3 by 0.9 in. Die pressures of 7 to 11 st/in^2 were evaluated. A midrange pressure, 9 st/in^2, was selected because 7 st/in^2 provided inadequate green density and compacts pressed at 11 st/in^2 often delaminated when ejected from the die.

SINTERING OF NICKEL-ALLOY-BONDED TiC SPECIMENS

Dewaxing, presintering, and sintering of specimens were performed consecutively in a vacuum sintering furnace, shown in figure 2. Heating was by graphite resistance elements, and samples were placed on a graphite tray inside a graphite felt box in the furnace chamber. The furnace was equipped with a digital controller for programming and control of the thermal cycle. For preliminary experiments on the 96 factorial experimental alloys, the following thermal treatment procedure was used:

1. Dewax step: Evacuate chamber to 50 × 10^{-3} mm Hg, backfill with hydrogen gas (industrial high-purity 99.95 pct H_2), and heat in flowing H_2 (1,076 L/h) from ambient temperature to 700° C at a rate of 10° C/min. Hold at 700° C for 30 min.
2. Presinter step: Increase from 700° to 850° C in flowing H_2 at a rate of 8° C/min. Hold at 850° C for 30 min. Increase at a rate of 13° C/min from 850° to 1,225° C. At 1,000° C, purge H_2 out of system with He (technical grade 99.995 pct He). Begin mechanical pump

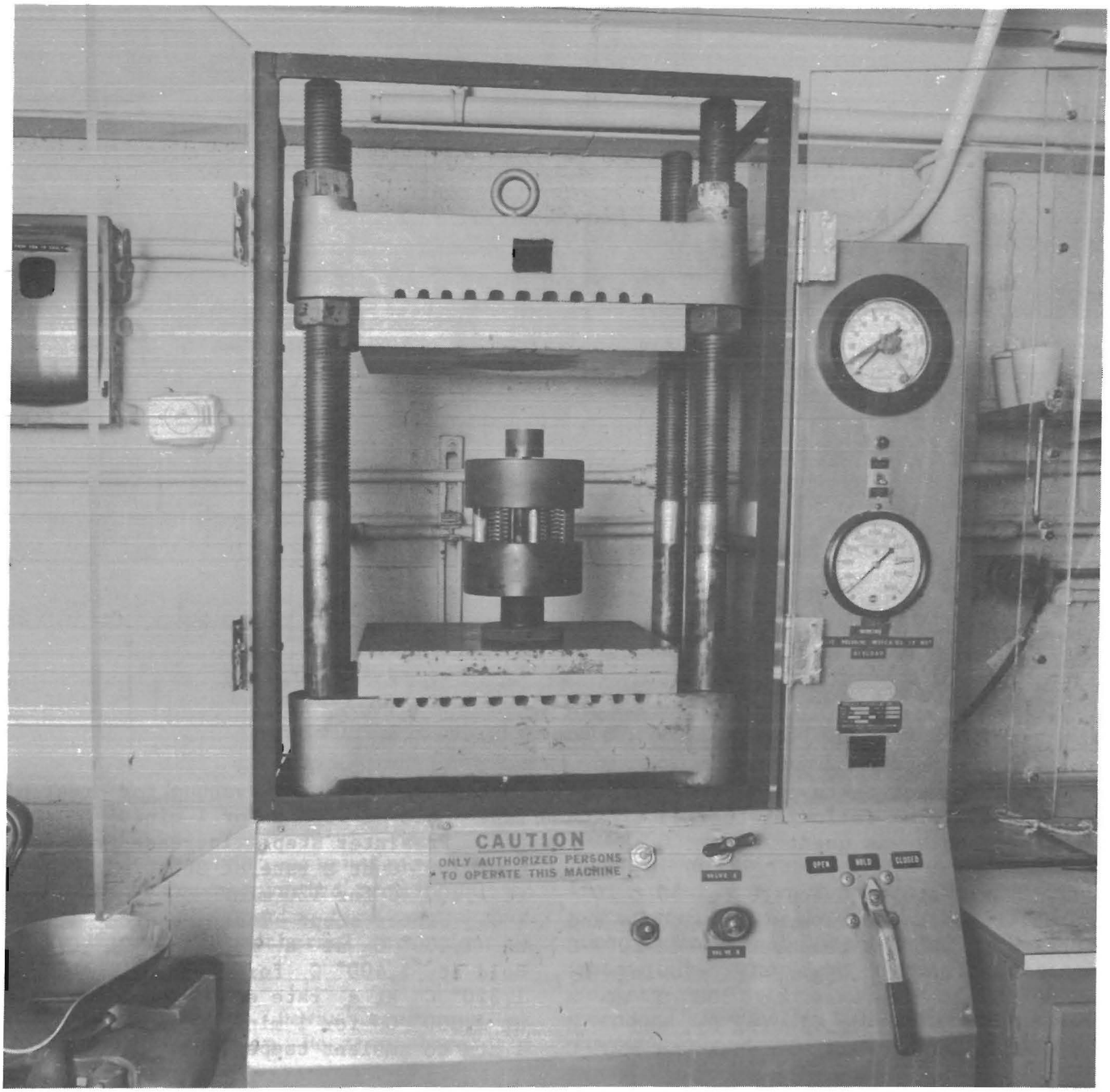

FIGURE 1.—Hydraulic press and die assembly for making powder compact specimens.

vacuum for rest of thermal treatment at 1,100° C. Hold at 1,225° C for 30 min.

3. Sinter step: Heat from 1,225° to 1,400° C at a rate of 3° C/min. Hold at 1,400° C for 60 min. Cool to 1,350° C and hold for 1 min. Quench in He at 1 atm to ambient temperature (approximately 2 h).

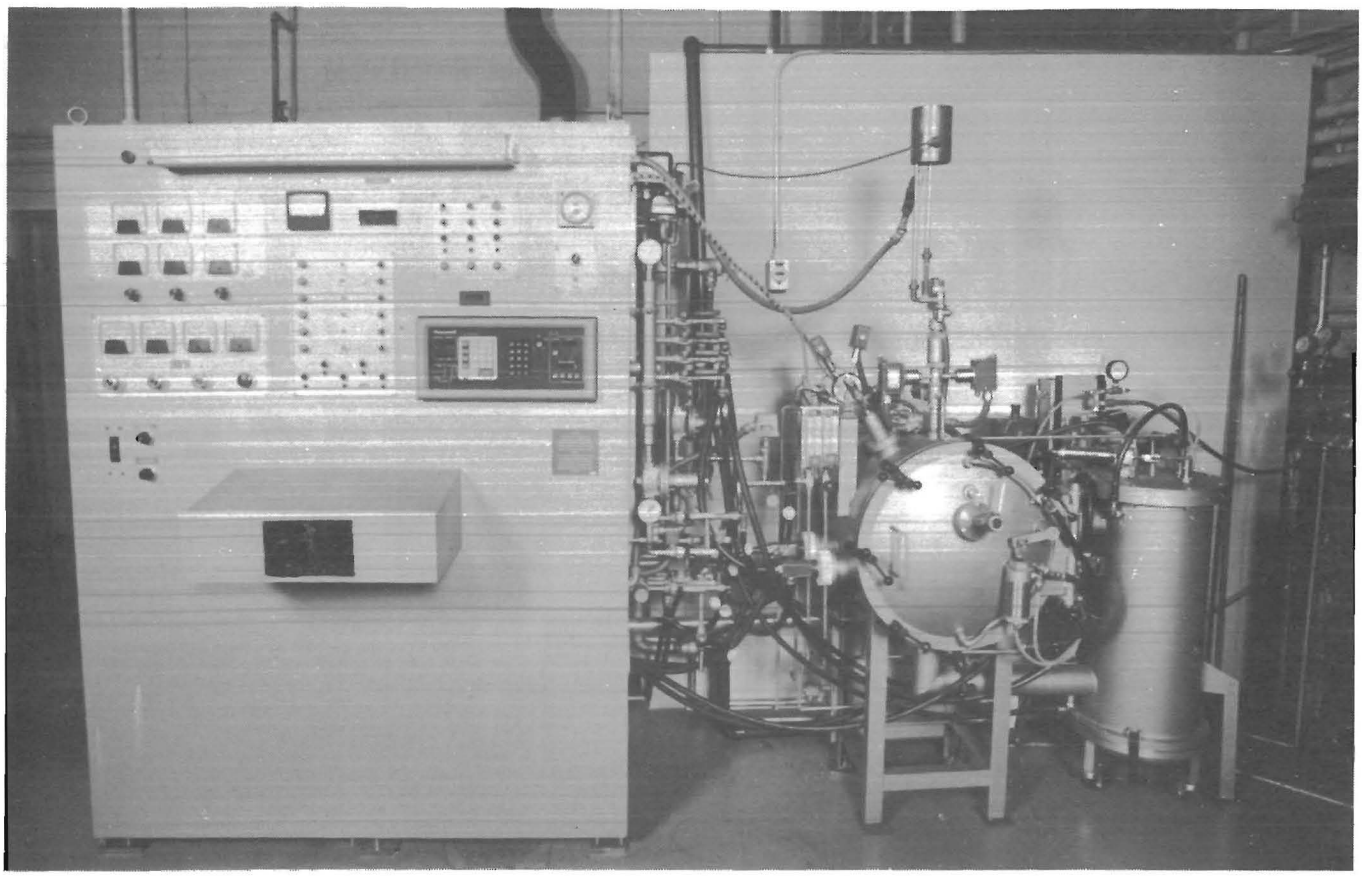

FIGURE 2.—Vacuum sintering furnace for Ni-alloy-bonded TiC materials.

For subsequent tests on quarter-replicate alloys, the following thermal treatment procedure was used:

1. Dewax step: Evacuate to 50×10^{-3} mm Hg; then backfill system with H_2 and heat from ambient temperature to 200° C at a rate of 10° C/min in flowing H_2 (1,076 L/h). Increase to 300° C at a rate of 5° C/min in flowing H_2, and hold at 300° C for 60 min. Increase to 350° C at a rate of 5° C/min in flowing H_2, and hold at 350° C for 30 min. Increase to 500° C at a rate of 10° C/min in flowing H_2, and hold at 500° C for 1 min. Purge H_2 out of system with He at 500° C. Increase from 500° to 600° C at 20° C/min. At 530° C, evacuate chamber to 200×10^{-3} mm Hg using a mechanical vacuum pump. Then evacuate using diffusion pump and mechanical forepump vacuum for rest of run. Hold at 600° C for 1 min.

2. Presinter step: Increase from 600° to 1,200° C at a rate of 20° C/min. Hold at 1,200° C for 120 min.

3. Sinter step: Increase from 1,200° to 1,400° C at a rate of 20° C/min. Hold at 1,400° C for 60 min. Cool to 1,350° C at a rate of 10° C/min. Hold at 1,350° C for 1 min. Quench in He at 1 atm to ambient temperature.

The thermal treatment as described for the preliminary 96 factorial alloys was used in the investigation to determine the effect of sintering time at 1,400° C on the mechanical properties of 60TiC-25Ni-Mo-W based alloys.

The effect of sintering temperature and time at final sintering temperature on

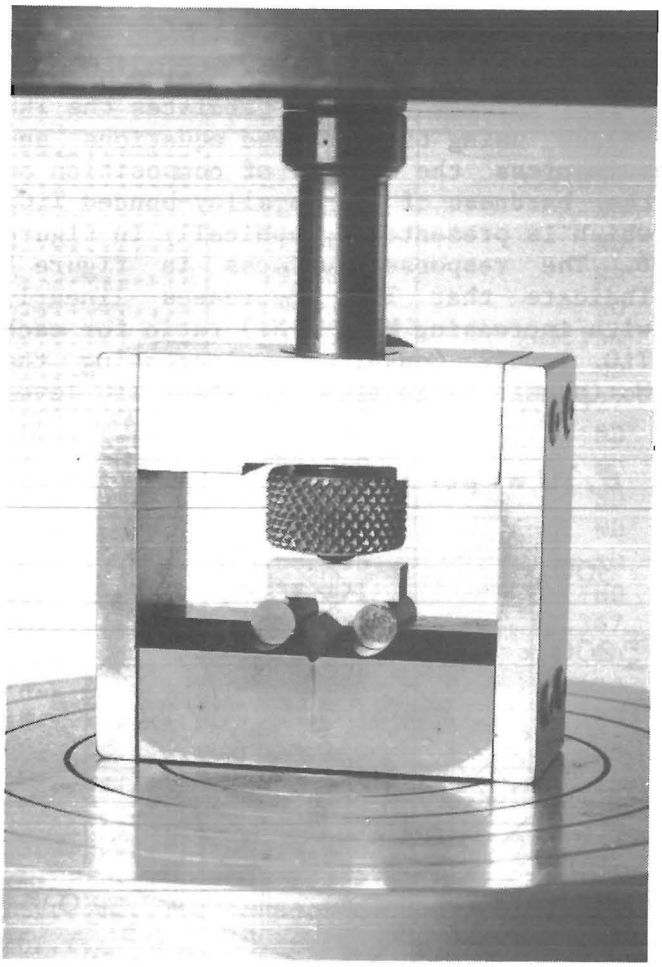

FIGURE 3.—TRS test apparatus.

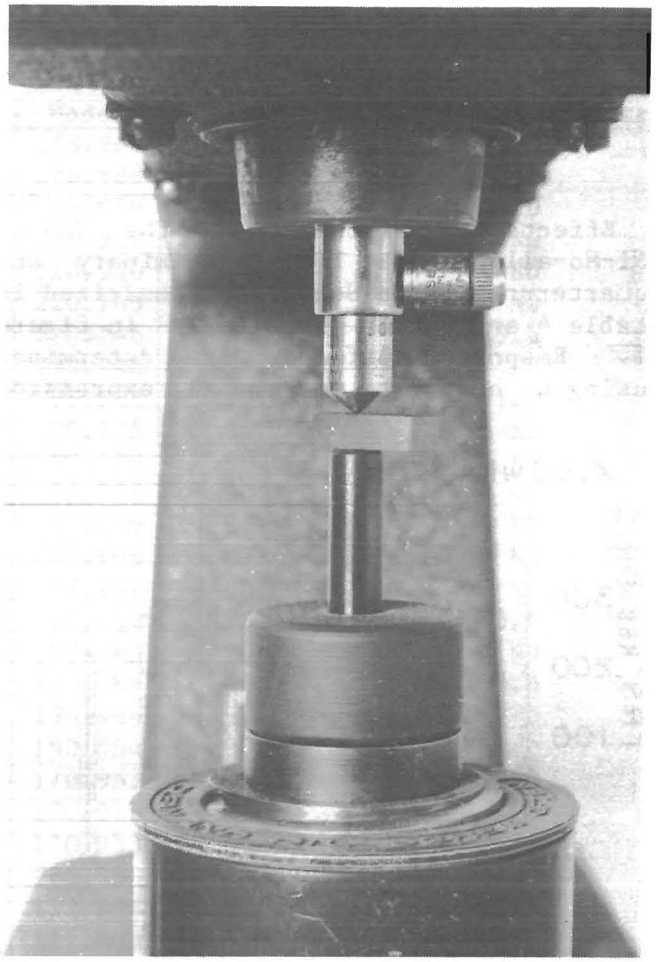

FIGURE 4.—Rockwell A hardness test apparatus.

the TRS of alloy 58 was studied. Sintering temperature was varied from 1,400° to 1,600° C, with sintering time varied from 30 to 120 min. The thermal treatment of specimens follows:

1. Dewax step: Dewax specimens at 354° C for 120 min in He atmosphere at a pressure of 0.8 atm.
2. Presinter step: Presinter at 850° C for 30 min and hold at 1,000° C for 120 min in flowing H_2 (1,076 L/h).
3. Sinter step: Hold at selected sintering temperature and time using a mechanical pump vacuum. Quench in He atmosphere until ambient temperature is reached.

MACHINING AND TESTING OF TRS SPECIMENS

Experimental alloy specimens for TRS and hardness tests were surface-ground to obtain the dimensions shown in American Society for Testing and Materials (ASTM) standard B-406-76 (16). Specimens were surface-ground using a 150-grit diamond wheel with soluble oil coolant. Finish dimensions were 0.200±0.01 by 0.250±0.01 by 0.750 in.

TRS and hardness (HRA) tests were conducted according to ASTM B-406-76 (16) and ASTM B-294-76 (17). Density measurements on sintered specimens were conducted according to ASTM B-311-58 (18). Figures 3 and 4 show TRS and hardness test apparatus, respectively.

RESULTS

EFFECT OF COMPOSITION ON TRS AND HARDNESS

Ni-Mo-Alloy-Bonded TiC

Effects of composition on the TRS of Ni-Mo-alloy-bonded TiC (preliminary and quarter-replicate data) are summarized in table 4 and shown graphically in figure 5. Response surfaces were determined using a best-fit polynomial expression with only significant terms being used by T-test values. Table 4 tabulates the TRS values using the derived equations and summarizes the effect of composition on the hardness of Ni-Mo-alloy-bonded TiC, which is presented graphically in figure 6. The response surfaces in figure 5 indicate that TRS decreases linearly with increasing Mo:(Mo+Ni) ratio for each TiC level. Conversely, increasing the Mo:(Mo+Ni) ratio at a constant TiC level

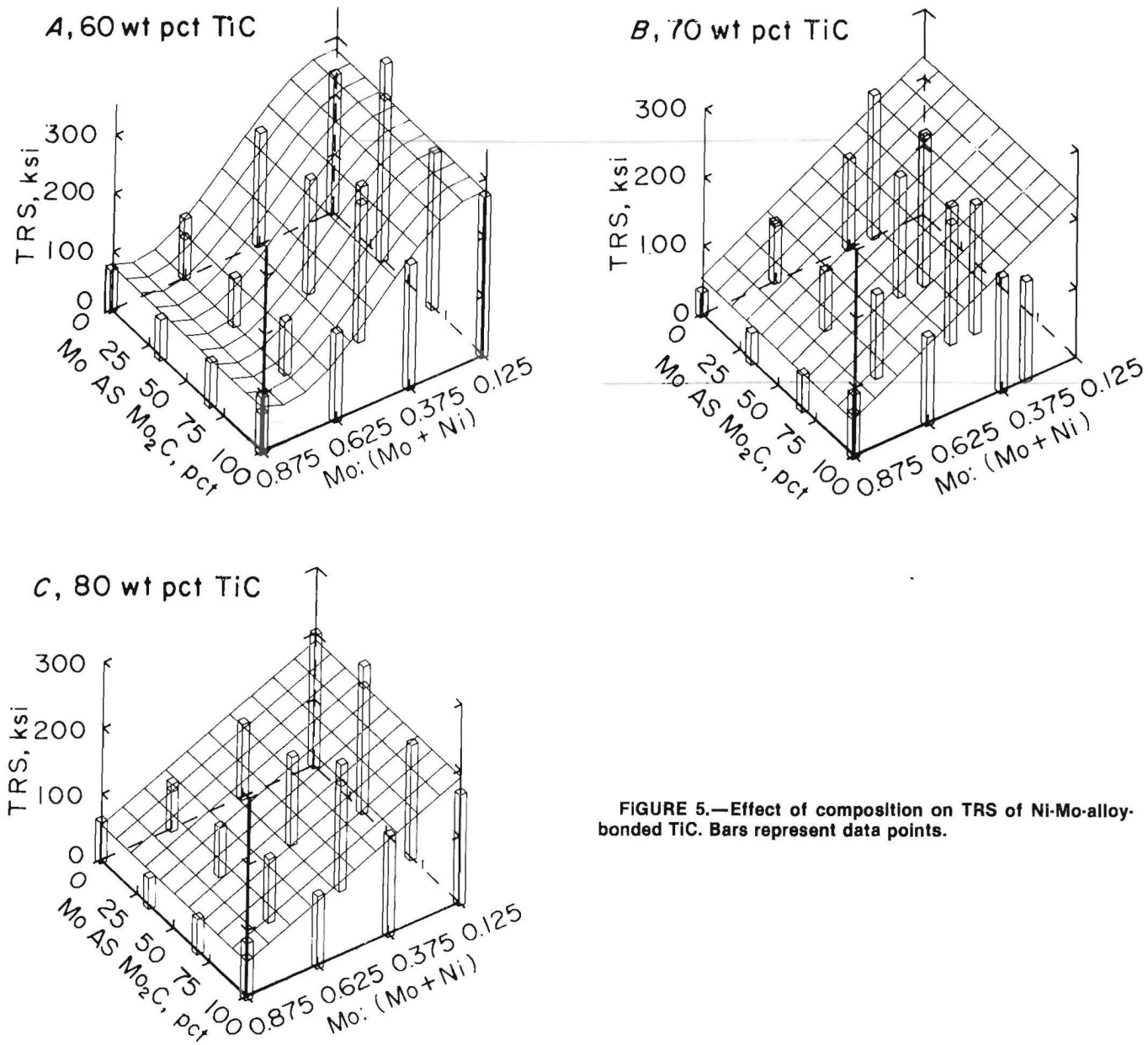

FIGURE 5.—Effect of composition on TRS of Ni-Mo-alloy-bonded TiC. Bars represent data points.

TABLE 4. - Effect of composition on TRS and hardness of Ni-Mo-alloy-bonded TiC

Alloy[1]	Transverse rupture strength, ksi			Hardness, HRA	
	Preliminary	Quarter replicate	Model	Preliminary	Quarter replicate
1.....	238.410	ND	278.989	85.6	ND
2.....	276.597	339.254	278.989	85.7	86.5
3.....	267.454	ND	278.989	85.5	ND
4.....	273.227	ND	278.989	85.2	ND
5.....	195.685	ND	220.746	89.1	ND
6.....	196.096	ND	220.746	89.1	ND
7.....	233.847	265.164	220.746	89.3	89.5
8.....	212.955	ND	220.746	89.2	ND
9.....	69.002	103.927	99.728	90.4	91.6
10.....	83.654	ND	99.728	90.7	ND
11.....	90.849	ND	99.728	90.0	ND
12.....	151.260	ND	99.728	90.2	ND
13.....	72.554	ND	77.109	87.5	ND
14.....	68.995	ND	77.109	87.8	ND
15.....	77.171	ND	77.109	88.6	ND
16.....	69.458	97.367	77.109	90.3	93.4
17.....	210.314	ND	190.664	90.2	ND
18.....	219.900	214.409	190.664	90.0	90.6
19.....	188.061	ND	190.664	90.3	ND
20.....	145.286	ND	190.664	90.3	ND
21.....	132.928	ND	170.953	90.1	ND
22.....	175.420	ND	170.953	90.6	ND
23.....	174.666	201.025	170.953	90.6	91.4
24.....	167.317	ND	170.953	90.8	ND
25.....	83.556	87.919	111.796	92.3	92.7
26.....	86.823	ND	111.796	91.1	ND
27.....	121.714	ND	111.796	91.5	ND
28.....	128.279	ND	111.796	90.7	ND
29.....	35.074	ND	52.640	87.8	ND
30.....	44.999	ND	52.640	ND	ND
31.....	54.008	ND	52.640	ND	ND
32.....	64.798	93.776	52.640	ND	93.0
33.....	202.557	ND	190.226	90.6	ND
34.....	191.190	223.869	190.226	89.7	90.5
35.....	174.107	ND	190.226	89.1	ND
36.....	167.716	ND	190.226	89.1	ND
37.....	115.128	ND	144.506	90.1	ND
38.....	134.064	ND	144.506	89.3	ND
39.....	159.710	191.818	144.506	90.9	91.2
40.....	153.296	ND	144.506	90.9	ND
41.....	60.912	72.670	98.783	ND	92.1
42.....	75.106	ND	98.783	89.0	ND
43.....	96.801	ND	98.783	88.4	ND
44.....	109.861	ND	98.783	90.5	ND
45.....	61.701	ND	53.060	ND	ND
46.....	46.824	ND	53.060	ND	ND
47.....	53.759	ND	53.060	ND	ND
48.....	60.620	85.284	53.060	ND	92.5

ND Not determined. [1]Alloy compositions are shown in table 1.

increases the hardness of the alloy, as shown in figure 6. The fraction of Mo added as Mo_2C did not have a significant influence on hardness and TRS.

TRS was dramatically decreased when the TiC content was increased from 60 to 80 wt pct (thus decreased Ni content) for alloys with Mo:(Mo+Ni) ratios of 1/8 to 3/8. At high Mo contents, Mo:(Mo+Ni) ratios of 5/8 or greater, TRS was independent of composition of the alloys, and strength values were less than 100 ksi. Hardness was significantly increased when the TiC content was increased from 60 to 70 wt pct (figures 6*A* and 6*B*, respectively). Based on results of the factorial experiment, a promising alloy composition was determined to be 59.37TiC-25Ni-5Mo-10.63Mo_2C (alloy 7), having values reported on quarter-replicate data of 265.164 ksi for TRS and 89.5 HRA for hardness. The best combination of TRS and hardness was for alloys containing 60TiC-25Ni-15Mo and 60TiC-25Mo-15Mo_2C.

Ni-Mo-W-Alloy-Bonded TiC

In this factorial experiment, the approach was (1) to improve properties by strengthening the Ni-Mo alloy binder by substituting W for part or all of the Mo, and (2) to promote toughness and ductility in the carbide phase by incorporating minor WC additions. Tungsten was a minor component, amounting to a maximum of 15 wt pct.

Results of the effect of composition on the TRS and hardness of Ni-Mo-W-alloy-bonded TiC (preliminary and quarter-replicate data) are summarized in table 5 and presented graphically in figures 7 and 8, respectively. Response surfaces shown were determined by a best-fit polynomial expression using only significant terms from T-test values. For alloys containing a nominal 60 wt pct TiC, TRS increased to a maximum for alloys containing two-thirds of the Mo and W added as carbides. Substitution of W for Mo did not have a significant influence on TRS. The strongest composition in the series based on 70 wt pct TiC was at a W:(W+Mo) ratio of 3/3 with all W added as WC. No clear maximum in the response surface for the series based on 80 wt pct TiC was indicated. As shown in figure 8, hardness increased significantly as the TiC content increased from 60 to 80 wt pct. Hardness did not vary significantly when W was substituted for Mo or when Mo and W were added as carbides up to W:(W+Mo) ratios of 5/8. When W was totally substituted for Mo for the series based on 60 and 70 wt pct TiC, hardness values decreased. Quarter-replicate data did not show this effect. Based on TRS

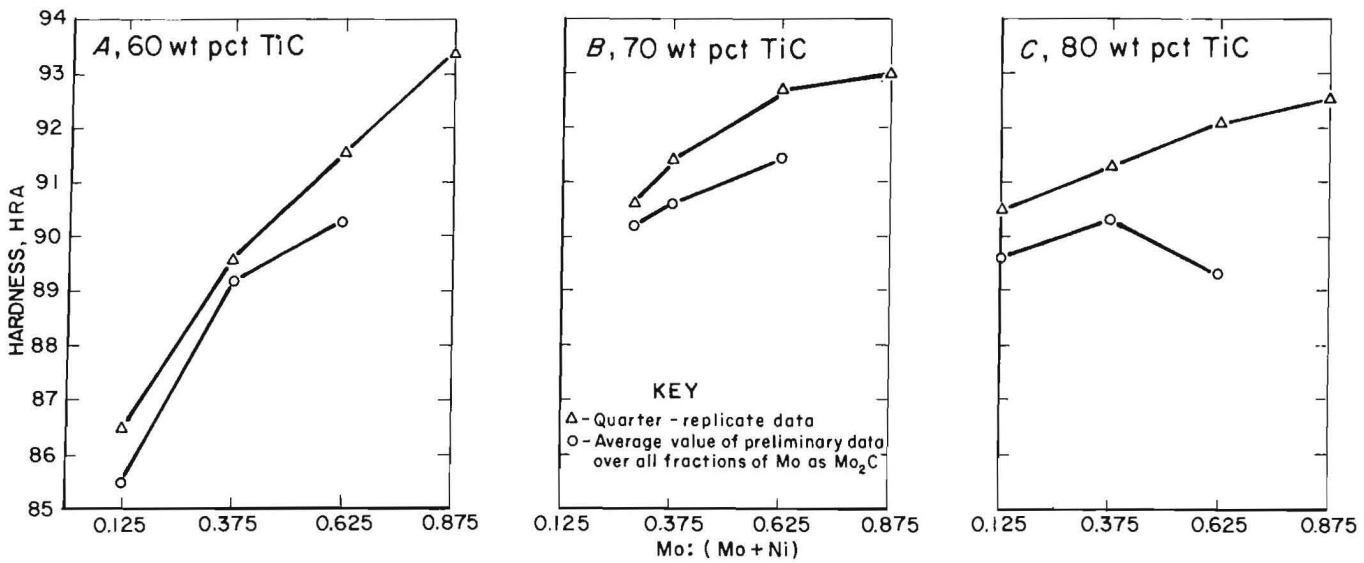

FIGURE 6.—Hardness as a function of Mo: (Mo + Ni) ratio for Ni-Mo-alloy bonded TiC.

TABLE 5. - Effect of composition on TRS and hardness of Ni-Mo-W-alloy-bonded TiC

Alloy[1]	Transverse rupture strength, ksi			Hardness, HRA	
	Preliminary	Quarter replicate	Model	Preliminary	Quarter replicate
49.....	191.233	ND	182.045	89.7	ND
53.....	150.851	175.881	182.045	89.7	89.6
57.....	189.269	ND	182.045	89.8	ND
61.....	205.130	ND	182.045	88.6	ND
50.....	201.296	ND	229.413	90.0	ND
54.....	227.921	ND	229.413	89.6	ND
58.....	224.744	242.753	229.413	89.2	89.5
62.....	243.928	ND	229.413	88.4	ND
51.....	219.056	255.499	244.107	89.6	89.8
55.....	240.162	ND	244.107	89.1	ND
59.....	290.013	ND	244.107	88.9	ND
63.....	222.187	ND	244.107	88.3	ND
52.....	206.155	ND	226.041	89.3	ND
56.....	212.894	ND	226.041	88.9	ND
60.....	228.622	ND	226.041	88.8	ND
64.....	181.252	ND	226.041	88.0	ND
65.....	112.445	ND	167.840	91.4	ND
66.....	187.718	ND	177.146	90.4	ND
67.....	193.193	216.979	186.480	90.4	91.3
68.....	219.685	ND	195.788	90.3	ND
69.....	178.008	154.329	181.370	90.7	90.8
70.....	214.581	ND	190.677	90.5	ND
71.....	193.914	ND	200.011	90.5	ND
72.....	183.151	ND	209.318	90.7	ND
73.....	206.730	ND	194.940	90.5	ND
74.....	216.831	198.643	204.248	90.0	90.8
75.....	218.124	ND	213.583	90.2	ND
76.....	228.343	ND	222.889	90.3	ND
77.....	232.273	ND	208.472	90.3	ND
78.....	242.070	ND	217.778	90.5	ND
79.....	210.561	ND	227.113	87.7	ND
80.....	207.331	227.698	236.420	89.2	90.7
81.....	162.191	ND	166.587	90.9	ND
82.....	164.736	ND	166.587	89.9	ND
83.....	156.877	188.053	166.587	90.2	92.2
84.....	165.405	ND	166.587	90.5	ND
85.....	177.261	160.240	174.374	91.2	91.7
86.....	156.917	ND	174.374	90.1	ND
87.....	184.842	ND	174.374	90.6	ND
88.....	176.630	ND	174.374	90.3	ND
89.....	182.080	ND	182.185	91.0	ND
90.....	188.536	204.891	182.185	90.4	92.0
91.....	176.049	ND	182.185	89.6	ND
92.....	178.386	ND	182.185	90.7	ND
93.....	178.400	ND	189.972	91.6	ND
94.....	201.111	ND	189.972	90.4	ND
95.....	193.792	ND	189.972	90.0	ND
96.....	178.510	190.685	189.972	90.6	92.2

ND Not determined. [1]Alloy compositions are shown in tables 1 and 2.

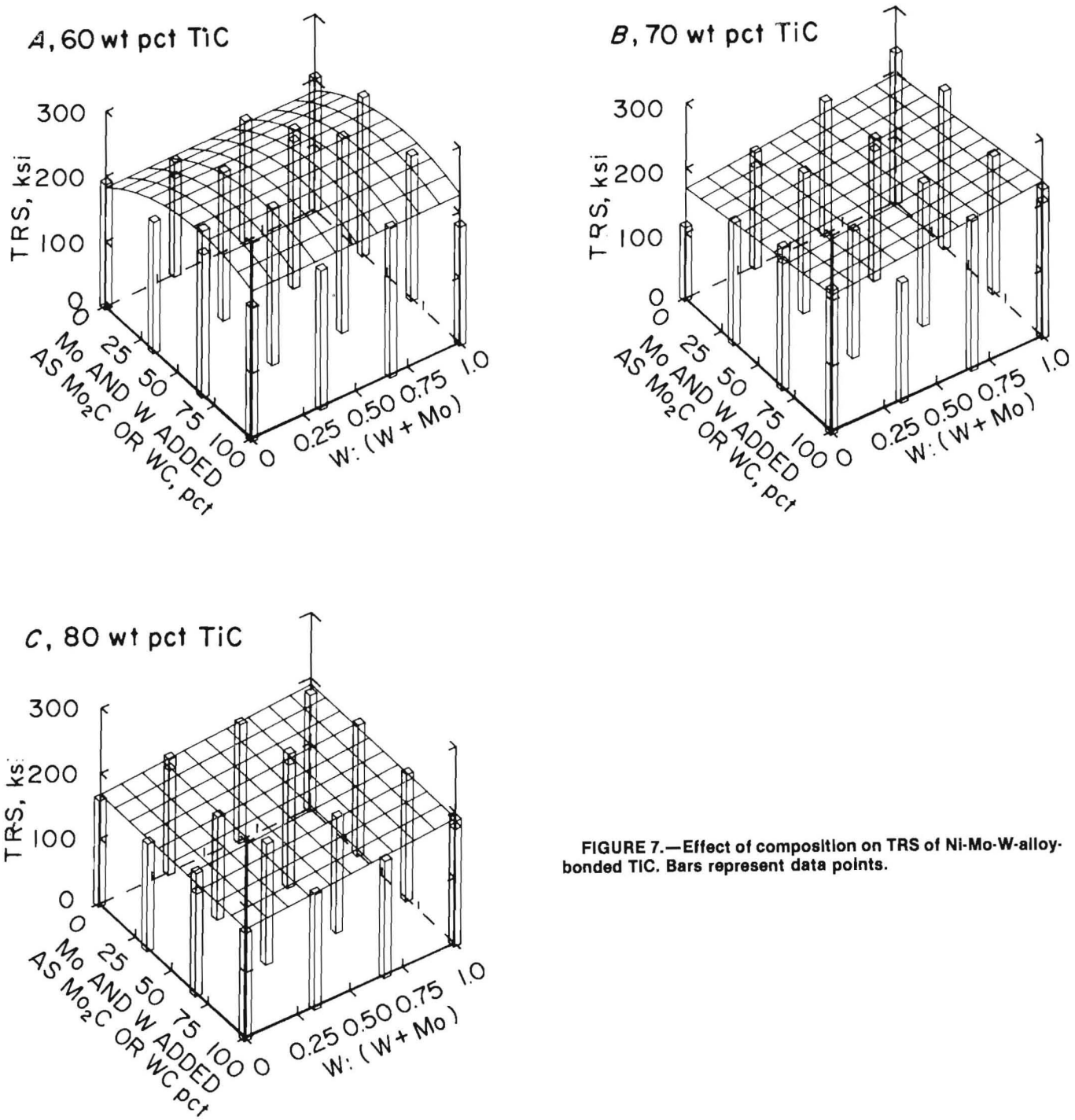

FIGURE 7.—Effect of composition on TRS of Ni-Mo-W-alloy-bonded TiC. Bars represent data points.

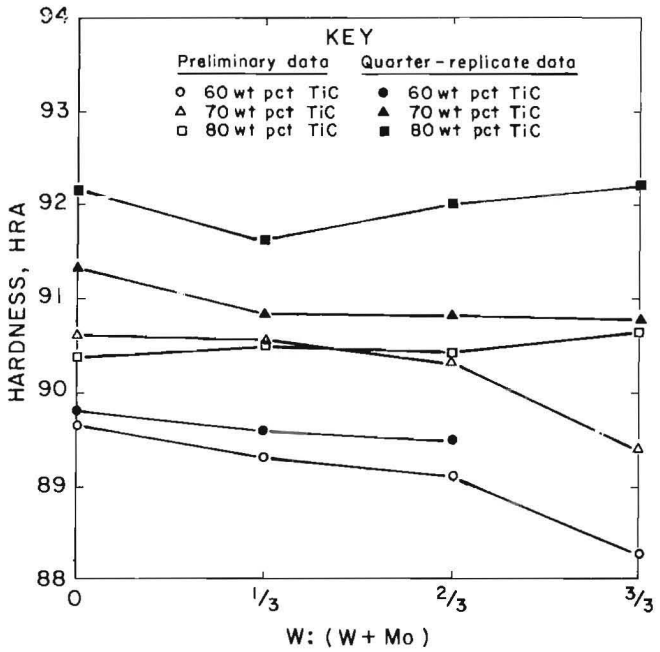

FIGURE 8.—Effect of substituting W for Mo on hardness of Ni-Mo-W-bonded TiC.

and hardness results, three compositions exhibited promising mechanical properties: alloys 51, 58, and 59. The compositions of these alloys are given in table 2, and their property values are given in table 6.

Comparison of Results for Preliminary and Quarter-Replicate Alloys

A comparison based on linear analysis of the TRS of preliminary and quarter-replicate alloys indicated that quarter-replicate alloy values were approximately 13 pct higher overall. A relationship between preliminary (P) and quarter-replicate (Q) TRS data was determined to be TRSQ = 21.31 + 1.015 (TRSP), where TRSQ and TRSP are expressed in units of ksi. A summary of preliminary and quarter-replicate data is given in table 7. Density values for these alloys were determined and are presented in table 7. Quarter-replicate Ni-Mo-alloy-bonded TiC alloys having Mo:(Mo+Ni) ratios of 5/8 and 7/8 were 1.4 HRA units higher than preliminary tests overall. Density data indicate that these alloys were an average of 0.25 g/cm^3 higher for quarter-replicate tests.

Quarter-replicate data on Ni-Mo-W-alloy-bonded TiC for the series based on 70 and 80 wt pct TiC were an average of 1 HRA unit higher and 0.15 g/cm^3 higher in density. This was attributed to improved milling and sintering procedures for the quarter-replicate alloys.

EFFECT OF SINTERING TEMPERATURE AND TIME

TRS, Hardness, and Lattice Parameters of 60TiC-25Ni-Mo-W Alloys

The effect of sintering time at 1,400° C on TRS and hardness is summarized in table 8 and presented graphically in figure 9. Results for alloy compositions with Mo and W added as elemental constituents indicate that (1) for sintering times up to 60 min, TRS remained unchanged, (2) hardness remained essentially constant for sintering times exceeding 30 min, (3) TRS was not affected by substituting W for Mo, and (4) hardness of Ni-W-bonded TiC was lower than that of alloys containing only partial substitution of W for Mo. For alloys containing Mo and W added as carbides, results indicate that (1) TRS decreased slightly when sintering times exceeded 60 min, (2) hardness of these alloys remained constant after sintering for 30 min, and (3) substituting W for Mo had no significant effect on TRS.

TABLE 6. - Mechanical properties of promising Ni-alloy-bonded TiC

Alloy[1]	Transverse rupture strength, ksi	Hardness, HRA	Alloy[1]	Transverse rupture strength, ksi	Hardness, HRA
7[2].....	265	89.5	58.....	243	89.5
51.....	255	89.8	59.....	290	88.9

[1]Alloy compositions are shown in tables 1 and 2.
[2]Alloy 7 is the same composition as alloy 51.

TABLE 7. - Comparison of TRS, hardness, and density of preliminary and quarter-replicate tests

Alloy[1]	Transverse rupture strength, ksi		Hardness, HRA		Density, g/cm^3	
	Preliminary	Quarter replicate	Preliminary	Quarter replicate	Preliminary	Quarter replicate
Ni-Mo-ALLOY-BONDED TiC						
2.....	276.6	339.3	85.7	86.5	6.01	6.01
7.....	233.9	265.2	89.3	89.5	6.02	6.04
9.....	69.0	103.9	90.4	91.6	5.86	6.05
16.....	69.5	97.4	90.3	93.4	5.76	6.06
18.....	219.9	214.4	90.0	90.6	5.60	5.72
23.....	174.7	201.0	90.6	91.4	5.63	5.73
25.....	83.6	87.9	92.3	92.7	5.59	5.73
32.....	64.8	93.8	ND	93.0	5.40	5.73
34.....	191.2	223.9	89.7	90.5	5.29	5.44
39.....	159.7	191.8	90.9	91.2	5.25	5.44
41.....	60.9	72.7	ND	92.1	5.02	5.43
48.....	60.6	85.3	ND	92.5	5.36	5.41
Ni-Mo-W-ALLOY-BONDED TiC						
51.....	219.1	255.5	89.6	89.8	6.01	6.04
53.....	150.8	175.9	89.7	89.6	6.12	6.14
58.....	224.7	242.8	89.2	89.5	6.21	6.23
64.....	181.3	ND	88.0	ND	6.29	ND
67.....	193.2	217.0	90.4	91.3	5.61	5.72
69.....	178.0	154.3	90.7	90.8	ND	5.80
74.....	216.8	198.6	90.0	90.8	5.73	5.85
80.....	207.3	227.7	89.2	90.7	5.77	5.90
83.....	156.9	188.1	90.2	92.2	5.23	5.44
85.....	177.3	160.2	91.2	91.7	5.35	5.53
90.....	188.5	204.9	90.4	92.0	5.34	5.52
96.....	178.5	190.7	90.6	92.2	5.40	5.54

ND Not determined. [1]Alloy compositions are shown in tables 1 and 2.

Results of X-ray diffraction studies are summarized in table 9 and presented in figure 10. The results indicate that the solid-solution mixed-carbide layer around TiC cores had completely formed within 0.5 min at 1,400° C, since lattice parameter measurements of the carbide phase remained unchanged for times exceeding 0.5 min. It was apparent that the heating cycle must be interrupted prior to 1,400° C to observe nonequilibrium conditions. Addition of Mo and W as carbides and complete substitution of W for Mo decreased the lattice parameter of the Ni binder phase. Results indicate that Ti, Mo, and/or W continue to be dissolved in the Ni phase for sintering times exceeding 0.5 min. Scanning electron microscopy using EDAX was used to determine the relative amounts of

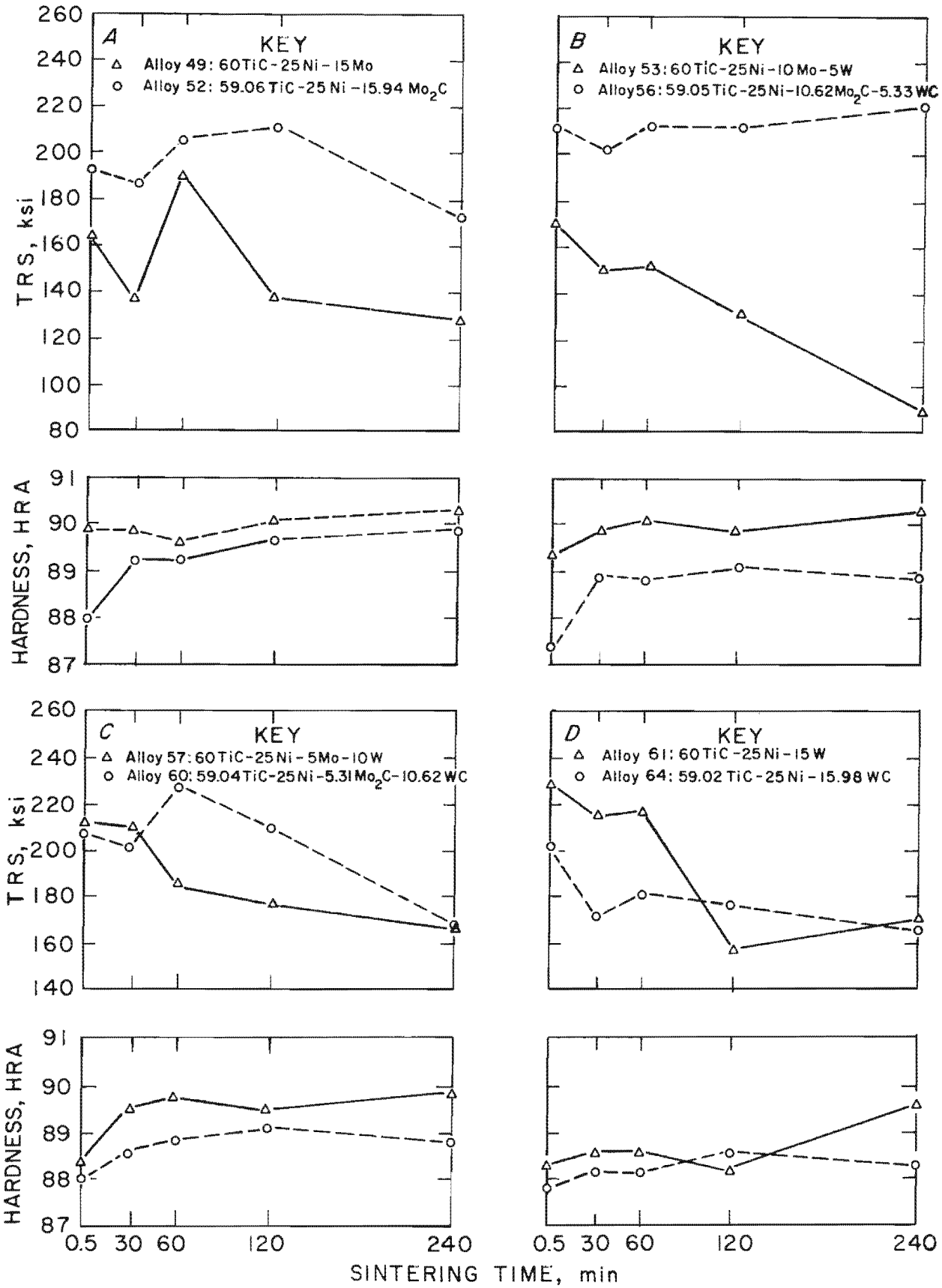

FIGURE 9.—Effect of sintering time at 1,400° C on TRS and hardness of 60TiC-25Ni-Mo-W alloys.

TABLE 8. - Effect of sintering time at 1,400° C on TRS and hardness of 60TiC-25Ni-Mo-W alloys

Alloy	Time at 1,400° C, min	Transverse rupture strength, ksi	Hardness, HRA	Alloy	Time at 1,400° C, min	Transverse rupture strength, ksi	Hardness, HRA
49.....	0.5	166±31	89.9	57.....	0.5	214±14	88.4
	30	138±40	89.9		30	212±12	89.5
	60	191±15	89.7		60	185±35	89.8
	120	139±12	90.1		120	177±13	89.5
	240	129±19	90.3		240	168±19	89.9
52.....	.5	193±11	88.0	60.....	.5	209± 9	88.0
	30	187±20	89.3		30	202±27	88.7
	60	207±26	89.3		60	228±32	88.8
	120	213±15	89.7		120	210±22	89.1
	240	172±25	89.9		240	170±12	88.8
53.....	.5	171±4	89.4	61.....	.5	229± 3	88.3
	30	151±34	89.9		30	214±16	88.6
	60	152±24	90.1		60	218±27	88.6
	120	131± 8	89.4		120	158± 2	88.2
	240	91±12	90.3		240	170±17	89.5
56.....	.5	212±15	87.3	64.....	.5	201±31	87.8
	30	199±4	88.9		30	172±25	88.1
	60	211± 7	88.8		60	181±41	88.1
	120	211±11	89.1		120	176±18	88.5
	240	222±13	88.9		240	165± 5	88.2

TABLE 9. - Lattice parameter measurements of carbide and matrix phases for sintering time at 1,400° C

Alloy	Sintering time, min	Lattice parameter of TiC, Å	Lattice parameter of Ni, Å	Alloy	Sintering time, min	Lattice parameter of TiC, Å	Lattice parameter of Ni, Å
49...	0.5	4.328±0.002	3.589±0.008	57...	0.5	4.329±0.002	3.590±0.004
	30	4.323± .004	3.580± .006		30	4.328± .002	3.583± .005
	60	4.323± .004	3.583± .005		60	4.326± .001	3.587± .003
	120	4.327± .002	3.586± .008		120	4.325± .001	3.567± .001
	240	4.327± .003	3.589± .008		240	4.324± .004	3.577± .006
52...	.5	4.330± .002	3.590± .005	60...	.5	4.329± .002	3.560± .005
	30	4.329± .002	3.578± .006		30	4.329± .002	3.568± .003
	60	4.331± .002	3.590± .008		60	4.329± .002	3.560± .007
	120	4.328± .002	3.587± .006		120	4.328± .002	3.560± .005
53...	.5	4.327± .002	3.589± .005	61...	.5	4.331± .002	3.592± .004
	30	4.318± .004	3.581± .010		30	4.330± .001	3.553± .009
	60	4.325± .002	3.582± .004		60	4.332± .001	3.559± .001
	120	4.326± .002	3.586± .006		120	4.330± .001	3.553± .006
	240	4.325± .002	3.578± .006		240	4.326± .001	3.572± .001
56...	.5	4.328± .002	3.564± .005	64...	.5	4.330± .001	3.557± .003
	30	4.328± .002	3.576± .002		30	4.334± .002	3.562± .001
	60	4.327± .002	3.572± .007		60	4.331± .002	3.556± .004
	120	4.333± .003	3.574± .002		120	4.331± .002	3.564± .007

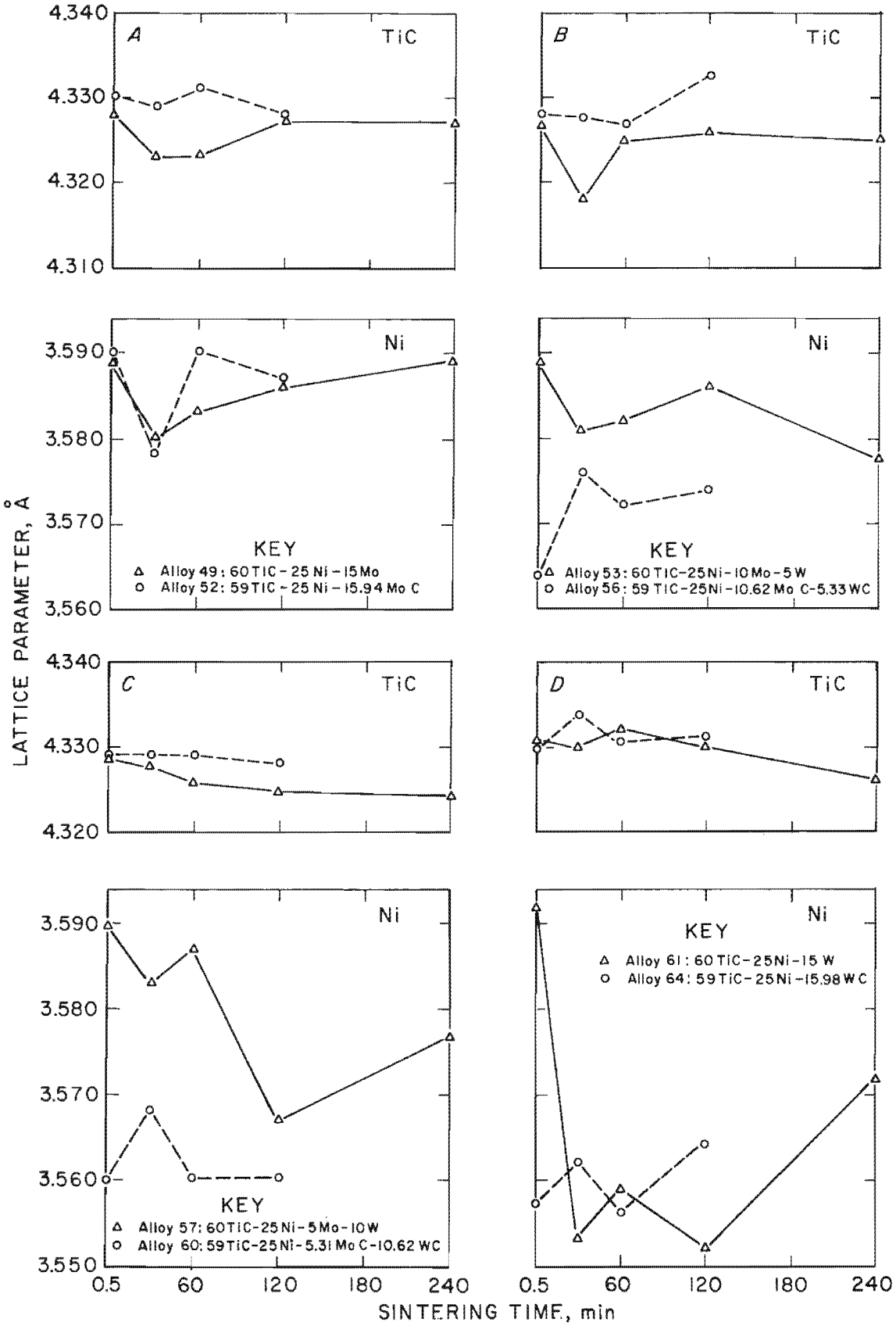

FIGURE 10.—Effect of sintering time at 1,400° C on the lattice parameter of cemented TiC alloys.

elements in the TiC cores, in the solid-solution mixed-carbide layer, and in the Ni binder phases. A scanning electron photomicrograph taken at X 10,000 (fig. 11) illustrates the typical microstructure of Ni-alloy-bonded TiC. Since the spatial resolution of the analyses was about 2 to 3 μm, there was some overlap between phases analyzed. Results are summarized in table 10. Supplementary to X-ray diffraction analyses, EDAX results indicated that (1) the carbide core was almost exclusively TiC, the rim was composed of a mixed carbide phase of Ti, Mo, and/or W, and small amounts of Ti, Mo, and/or W were dissolved in the nickel binder, and (2) the mixed carbide phase had completely formed within 0.5 min at 1,400° C.

TABLE 10. - EDAX elemental analysis results of sintering time at 1,400° C, weight percent

Alloy	Time at 1,400° C, min	TiC core				Rim				Binder			
		Ti	Ni	Mo	W	Ti	Ni	Mo	W	Ti	Ni	Mo	W
49....	0.5	99.48	0.52	0	0.00	77.59	4.41	16.49	1.51	14.22	83.74	1.29	0.75
	60	99.16	.43	0	.41	76.60	2.41	19.72	1.54	17.60	81.50	.45	.44
52....	60	96.57	.94	2.18	.30	67.31	9.97	21.88	.83	9.09	87.16	1.66	2.08
53....	.5	98.84	.78	0	.38	70.94	5.20	9.78	14.58	12.49	85.19	.53	1.80
	60	99.23	.63	0	.14	74.91	1.55	14.86	8.67	16.31	81.41	.83	1.46
56....	60	99.41	.59	0	0	77.97	2.80	11.19	8.04	15.85	80.16	1.96	2.03
57....	.5	98.84	.50	0	.66	64.00	16.58	5.72	13.70	11.99	86.91	.26	.84
	60	98.98	.50	0	.52	62.23	16.22	4.95	16.60	12.74	86.03	.42	.81
60....	60	96.85	1.80	0	1.35	68.28	10.21	5.25	16.26	29.40	57.29	3.66	9.65
61....	.5	99.35	.65	0	0	73.12	1.32	0	25.55	11.46	87.05	0	1.49
	60	97.36	1.87	0	.77	70.11	7.01	0	22.88	12.28	85.26	0	2.45
64....	60	98.38	.75	0	.86	67.32	1.79	.35	30.54	9.36	88.94	0	2.19

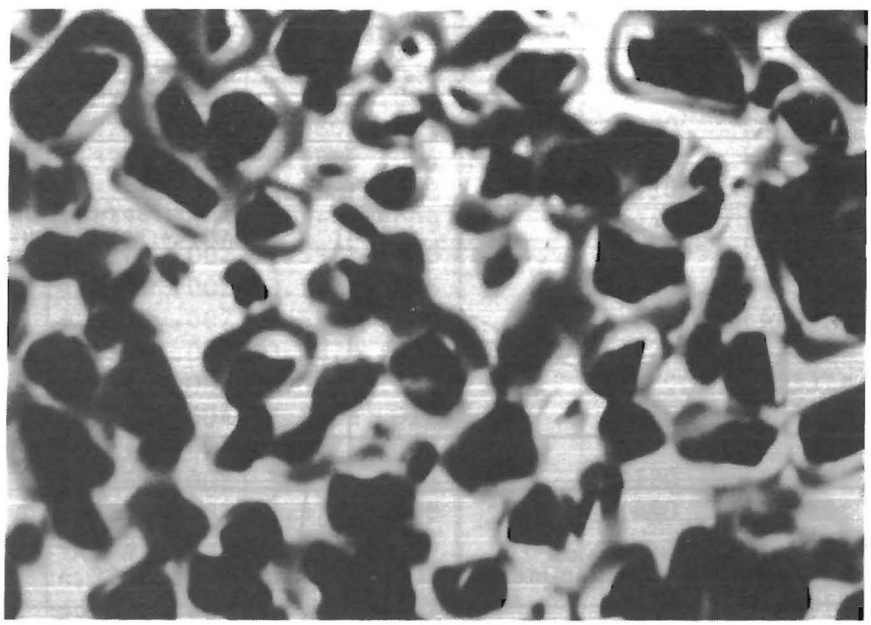

FIGURE 11.—Scanning electron photomicrograph showing typical microstructure of Ni-alloy-bonded TiC specimen (X 10,000).

TABLE 11. - Effect of sintering time at 1,400°, 1,500°, and 1,600° C on TRS of alloy 58[1]

Temp, °C	Time, min	Transverse rupture strength, ksi	Temp, °C	Time, min	Transverse rupture strength, ksi	Temp, °C	Time, min	Transverse rupture strength, ksi
1,400	30	ND	1,500	30	230.1±59.5	1,600	30	191.6±58.6
	60	234.8±45.0		60	160.4±27.3		60	176.2±38.5
	120	232.6±41.5		120	122.0±16.8		120	135.2±19.4

ND Not determined. [1]Composition: 59.68TiC-25Ni-3.33Mo-1.77Mo_2C-6.67W-3.55WC.

TRS of Alloy 58

The effect of sintering temperature and time at final sintering temperature on the TRS of alloy 58 was determined. Specimens for evaluation were sintered at 1,400°, 1,500°, and 1,600° C, with sintering time varied from 30 to 120 min. Results are summarized in table 11 and presented graphically in figure 12. As shown, TRS decreased as both sintering temperature and time increased. Results indicate that sintering temperature should be kept at or below 1,400° C, and sintering time should be kept at less than 60 min.

DISCUSSION OF RESULTS

The effect of composition and processing variables on the TRS and hardness of Ni-alloy-bonded TiC were investigated. The principal characteristics of a cutting tool are its toughness and wear resistance, indicated by TRS and hardness, respectively. Strength values decreased linearly as the Mo:(Mo+Ni) increased for Ni-Mo-alloy-bonded TiC at a given TiC content, whereas hardness increased linearly. The TiC content of the alloy had a significant effect on the mechanical properties of the materials. For the Ni-Mo-W-alloy-bonded TiC factorial experiment, hardness of the materials was not significantly affected by substituting W for Mo or by whether the Mo and W additions were as elemental constituents or as carbides. TRS for the series based on 60 wt pct TiC was dependent on the fraction of Mo and W added as carbide constituents. For the series based on 70 wt pct TiC, the TRS was raised by increasing the W:(W+Mo) ratio and by adding Mo and W as carbides. No distinct relationship between TRS and composition variables was apparent for the series based on 80 wt pct TiC. Sintering temperature and time at final sintering temperature had a significant influence on the mechanical properties of Ni-alloy-bonded TiC.

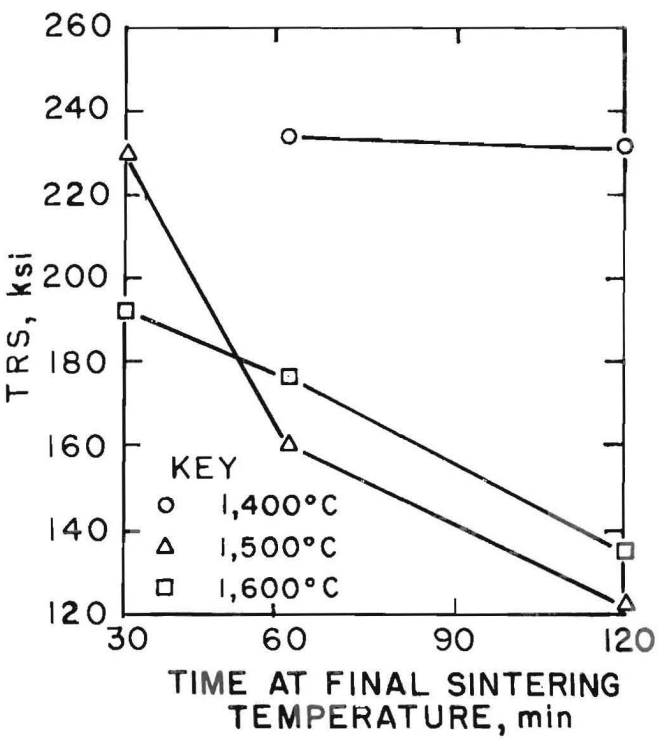

FIGURE 12.—Effect of sintering time at 1,400°, 1,500°, and 1,600° C on TRS of alloy 58 (59.68TiC-25Ni-3.33Mo-1.77Mo_2C-6.67W-3.55WC).

EFFECT OF COMPOSITION ON TRS AND HARDNESS

Ni-Mo-Alloy-Bonded TiC

The effect of composition on the TRS and hardness of Ni-Mo-alloy-bonded TiC was investigated for three series of alloys based on 60, 70, and 80 wt pct TiC. Results showed that increasing the Mo:(Mo+Ni) from 1/8 to 7/8 drastically reduced the strength of the material at a constant TiC content. Additions of Mo to Ni-bonded TiC formed solid-solution layers, (Ti,Mo)C, around essentially Mo-free TiC grains. Beneficial effects of Mo additions were enhanced wettability and suppressed TiC grain growth, which in turn increased the toughness and hardness of the material (8-9). Moskowitz (9) demonstrated that for TiC-Ni-Mo_2C materials, TRS increased linearly by variation of Ni content up to 40 vol pct at a constant Mo-TiC ratio of 1/7. Hardness decreased linearly as the volume percent binder increased. A similar relationship was demonstrated in the present study. Hardness decreased dramatically in a linear fashion as the Mo:(Mo+Ni) decreased from a value of 7/8 to 1/8 at a constant TiC content. As previously stated, TRS in turn was significantly increased as the Mo:(Mo+Ni) ratio decreased. Results obtained are compatible with those reported by Moskowitz (9) and Nishigaki (12) on the hardness and TRS of Ni-Mo-alloy-bonded TiC materials. Quarter-replicate data were shown to have higher hardness and TRS. This was ascribed to improved milling and heat treatment procedures. Milling with acetione as a solvent resulted in improved wax distribution in pressed specimens, thus avoiding wax pockets that could result in pores upon heat treatment. Hardness values for preliminary alloys at an Mo:(Mo+Ni) ratio of 7/8 were not taken because of insufficient densification and low strength. Quarter-replicate tests of these alloys showed that improved densification can be ascribed to an improved milling and heat treatment cycle.

Based on TRS and hardness test results, a composition of 59.37TiC-25Ni-5Mo-10.63Mo_2C (alloy 7) exhibited the most promising mechanical properties. Values for quarter-replicate tests of 265,164 psi and 89.5 HRA for TRS and hardness were exhibited by alloy 7. In comparison, a 66.9TiC-22.5Ni-10.6Mo_2C composition for C-5 cutting tool material applications was reported to exhibit 275,000 psi and 90.6 HRA for TRS and hardness, respectively (13-14). It was estimated by test results that compositions with TiC contents between 60 and 70 wt pct, Mo:(Mo+Ni) ratios between 1/8 and 3/8, and two-thirds of the Mo added as carbide would have suitable mechanical properties for further investigation.

Ni-Mo-W-Alloy-Bonded TiC

The effect of composition on the TRS and hardness of Ni-Mo-W-alloy-bonded TiC was determined. The objective of this factorial experiment was to improve properties by strengthening the carbide and binder phases by W addition by a solid-solution strengthening mechanism. Microstructural development similar to that of TiC-Ni-Mo has been reported for TiC-Ni-W alloys (19).

Results indicate that for alloys containing 60 wt pct TiC, TRS reached a maximum at a 2/3 fraction of Mo and W added as carbides. Substituting W for Mo did not influence the TRS of these alloys. For the series based on 70 wt pct TiC, TRS was increased by increasing the W:(W+Mo) ratio and adding Mo and W as carbides. No maximum in TRS was observed for alloys based on 80 wt pct TiC. The hardness of Ni-Mo-W-alloy-bonded TiC alloys investigated was affected primarily by their TiC content. Increasing the TiC content, thus reducing the amount of binder, increased the hardness of the materials. Similar to Ni-Mo-alloy-bonded TiC, quarter-replicate tests also exhibited higher hardness and strength. For 60, 70, and 80 wt pct TiC series, Ni content was held at 25, 18.75, and 12.5 wt pct, respectively. Therefore, the binder composition was varied by Mo and W contents and the fraction of these constituents added as carbides. From the TRS results, less change in values was observed in comparison to the Ni-Mo-alloy-bonded TiC factorial experiment,

since Ni content was varied dramatically in the latter experimental design.

Based on TRS and hardness test results, the most promising alloys were alloys 51, 58, and 59. Compositions and properties are summarized in tables 2 and 6, respectively. Since the compositions substituting W for Mo had similar strength and hardness values, the value of W additions could only be shown by tool performance in machining tests.

EFFECT OF PROCESSING VARIABLES ON TRS AND HARDNESS OF Ni-ALLOY-BONDED TiC

Sintering temperature and soak time at final sintering temperature were determined to have a significant effect on TRS and hardness of Ni-alloy-bonded TiC. Results indicate that for Ni-alloy-bonded TiC when Mo and/or W were added as elemental constituents, TRS decreased after sintering at 1,400° C for periods in excess of 60 min. When Mo and/or W were added as carbide constituents to Ni-alloy-bonded TiC, TRS decreased slightly for periods greater than 60 min at 1,400° C. Hardness values remained unchanged after sintering for 30 min at 1,400° C for all Ni-alloy-bonded TiC compositions investigated. Results of X-ray diffraction studies showed that the mixed-carbide solid-solution layer had formed within 0.5 min at 1,400° C. Elements such as Ti, Mo, and W appeared to continue dissolving in the Ni binder phase for sintering in excess of 0.5 min at 1,400° C, since the lattice parameter of the Ni phases tended to decrease after 0.5 min at 1,400° C. A decrease in the lattice parameter of Ni was observed when Mo and W were added as carbide constituents, and the lattice parameter of Ni was significantly decreased for a TiC-Ni-W composition compared to a TiC-Ni-Mo composition. The resulting formation of the solid-solution layer around TiC grains was anticipated. Snell (11) reported that the microstructural development of the mixed-carbide phase in a TiC-24Mo-15Ni composition took place between 600° and 1,400° C. Lindau (19) reported that the microstructural development of Ni-W-bonded TiC followed a development similar to that of Ni-Mo-bonded TiC.

The effect of sintering temperature and time at final sintering temperature on the TRS of alloy 58 was studied. Results indicate that as sintering temperature increased from 1,400° to 1,600° C, TRS was dramatically reduced. Grain growth of TiC was observed in specimens sintered at 1,600° C. Extending the soak time at final sintering temperature also decreased TRS. These results are compatible with work reported by Snell (11) on the effect of sintering temperature on the TRS of TiC-24Mo-15Ni alloy. Grain growth was reported to be due to the growth of the mixed-carbide phase around TiC grains, which affected the fracture mode of the materials. At a grain size of 2 to 4 μm, fracture was reported to change from intergranular to transcrystalline through the carbide phase, and lower strengths were observed. TiC grain size of experimental compositions sintered at 1,400° C for 60 min was about 1 to 2 μm. Therefore, sintering of the Ni-alloy-bonded TiC compositions investigated should be kept at 1,400° C and should not exceed 60 min for good mechanical properties.

SUMMARY AND CONCLUSIONS

Studies were conducted to determine the effects of composition and processing variables on the TRS and hardness of Ni-alloy-bonded TiC. Specimens for evaluation were prepared by a powder metallurgy method of cold pressing and sintering. Compositions were prepared by milling constituent powders with grinding media, milling solvent, and a pressing wax. The heat treatment cycle provided holds at 300° to 700° C for wax removal, 1,200° C for presintering, and 1,400° C for liquid-phase sintering. The effects of sintering temperature and time at final sintering temperature on the TRS and hardness of Ni-alloy-bonded TiC were studied.

Two factorial experiments were designed to determine the effect of composition on the TRS and hardness of Ni-alloy-bonded

TiC. Variation of the Mo:(Mo+Ni) ratio and the fraction of Mo added as Mo_2C for three levels of TiC showed that--

1. TRS was decreased by increasing the Mo:(Mo+Ni) ratio at a constant TiC content.
2. TRS was decreased by increasing the TiC content from 60 to 80 wt pct TiC for materials with Mo:(Mo+Ni) ratios between 1/8 and 3/8. TRS was essentially independent of TiC content for alloys with Mo:(Mo+Ni) ratios between 5/8 and 7/8.
3. The TiC content of the composition had the most significant effect on increasing hardness between 60 and 70 wt pct TiC. No increase in values of 80 wt pct TiC series alloys was apparent.

Reported strength and hardness values were in general agreement with those reported in the literature for TiC-Ni-Mo compositions. In the second factorial experiment, substitution of W for Mo and variation of the fraction of Mo and W added as carbide constituents for a Ni:(Mo+W) ratio of 5/3 at three levels of TiC content showed that--

1. TRS was optimized for alloys containing two-thirds of the Mo and W added as carbide constituents, and no effect was apparent when substituting W for Mo in the series based on 60 wt pct TiC.
2. For the series based on 70 wt pct TiC, TRS was maximum at a W:(W+Mo) ratio of 1 with all the W added as WC.
3. No distinct maximum in strength was observed for the series based on 80 wt pct TiC.
4. Hardness of the material was significantly increased by increasing the TiC content, thus reducing the Ni-alloy binder content. With an increase in TiC content, TRS was reduced.

Based on the TRS and hardness results, alloys 51 (same as 7), 58, and 59 were determined to have the best combination of properties for the alloys investigated. Compositions, in weight percent, are alloy 51, 59.37TiC-25Ni-5Mo-10.63Mo_2C; alloy 58, 59.68TiC-25Ni-3.33Mo-1.77Mo_2C-6.67W-3.55WC; and alloy 59, 59.35TiC-25Ni-1.67Mo-3.54Mo_2C-3.33W-7.1WC.

Sintering temperature and time at final sintering temperature were determined to have an influential effect on the TRS and hardness of Ni-alloy-bonded TiC. Studies on 60TiC-25Ni-Mo-W compositions sintered at 1,400° C between 0.5 and 240 min showed that--

1. TRS was reduced for sintering times exceeding 60 min for compositions containing Mo and W added as elemental constituents, and was slightly reduced when Mo and W were added as carbide constituents.
2. Hardness values were essentially unchanged for sintering times greater than 30 min.
3. The mixed-carbide solid-solution layer around TiC grains had already formed for specimens sintered at 1,400° C for 0.5 min, since the lattice parameter of the carbide phase was constant for sintering times investigated.
4. Substituting W for Mo and adding Mo and W as carbide constituents resulted in a significant decrease in the lattice parameter of the binding phase.
5. Substituting W for Mo did not have a significant effect on the hardness and TRS of compositions studied.

Studies on alloy 58 to determine the effect of sintering temperature and time at final sintering temperature on the TRS showed that--

1. Increasing sintering temperature from 1,400° to 1,600° C decreased the TRS of the composition. Grain growth was observed in specimens sintered at 1,600° C.
2. Sintering times exceeding 60 min at final sintering temperature reduced the TRS of the material.

Results of this preliminary study show that alloys 51, 58, and 59 have the best combination of TRS and hardness. Processing procedures for quarter-replicate alloys indicate that those procedures should be adopted for subsequent evaluations. An objective of subsequent tests will be to increase toughness of these base compositions by solid-solution

strengthening additions in order to obtain compositions that meet or exceed properties reported in the literature for C-5-grade materials. Since substitution of W for Mo, as in alloys 58 and 59, gave similar strength and hardness values, beneficial effects of this substitution would only be delineated by machining test comparisons. Mechanical property values give only a good indication of the applicability of a composition as a cutting tool material. Final evaluation of compositions for substitute materials for conventional C-5-grade Co-bonded WC tools would depend on the materials' performance in machining test studies that are currently underway.

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