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CAST IRON BASE HEAT-RESISTANT ALLOYS

R. W. Kraft R. A: Flinn

Project 2313

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#### SUMMARY

This project was initiated on October 1, 1954, with two principal objectives in mind:

- 1. To develop a heat-resistant alloy for use in the rotor of the automotive gas turbine in place of the present expensive cobalt-base material. Minimum mechanical properties of 30,000 psi for 100-hour rupture strength at 1500°F and 70,000 psi tensile strength with 10% elongation in the room temperature tensile test were specified. Strategic alloy content was to be kept to a minimum.
- 2. To develop a production technique more economical than the investment-casting process now used.

Progress on objective 1 within the past year has consisted of:

- 1. Procurement of equipment and training of personnel in investment-casting procedures.
- 2. Satisfactory development of molding, melting, and pouring techniques as measured by the fact that properties obtained on standard materials produced at the University have equalled or exceeded commonly accepted industrial standards.
- 3. Substantial progress on a study of the effects of specific alloy additions on the casting characteristics, microstructure, and stress-rupture properties of an 18% Ni, 18% Cr, iron-base alloy. It is hoped that this investigation will point out why elements are added to heat-resistant alloys and how they become effective in enhancing properties.

Progress on objective 2 has centered about the shell-molding process. A test-bar pattern has been prepared and satisfactory castings have been produced. This process is inherently many times cheaper than the investment process, and it appears from preliminary experiments that it is easier to make clean, sound bars with this method. Some difficulties with surface quality have occurred and probably for this reason, the ductility of standard materials is somewhat less than comparable investment cast bars. These problems are thought to be mainly mechanical in nature, the primary difficulty being non-uniform flow characteristics of the sand mixture with resulting unhomogeneous

shells. Since this problem is not considered serious, experiments will be begun in the near future on a prototype casting.

The body of this report follows the same outline as this introduction:

Part I - Alloy Development Program.

Part II - Casting Processes.

Part III - Future Work.

Appendix - Tables and Illustrations

#### PART I. ALLOY DEVELOPMENT PROGRAM

### PROCEDURES

Mold Design.—Two basic concepts were involved in developing the investment mold design shown in Fig. 1; the desirability of smooth, nonturbulent metal flow, and the need for directional solidification in the gage length of the test bars to promote soundness.

A bottom-gating system was decided upon as being the most efficient method for producing nonturbulent flow. As the mold is inverted to the upright position (Fig. 3), the liquid metal flows down the sprue, out to the torus, and up into the test bars. The only turbulence present is that which is caused by the irregularities of the mold itself. This technique was thought to be superior to the system in general use in which hot metal is introduced directly into the risers near the end of the pour with the idea of putting the hottest metal into the risers. In the latter system, metal usually goes down the wrong sprue first, to a greater or lesser extent, thus producing cold shuts or very turbulent conditions with entrapment of slag or oxides because of two streams of metal traveling in opposite directions.

Directional solidification, the second factor, is accomplished by means of the large, individual blind risers on the upper end of each specimen. The purpose of the V-shaped depression in the risers is to allow atmospheric pressure to penetrate through the skin (which is formed first) and thus enhance the effectiveness of the riser. Early experiments were conducted with different design test bars, viz., two different designs, each with a length-to-diameter ratio of 2:1 instead of 4:1 (Figs. 11 and 12). Data presented in an earlier report showed that it was not necessary to use these nonstandard bars in order to obtain sound castings, so their use has been discontinued. These other designs could, however, provide a convenient method for evaluating different cooling rates and/or grain sizes, and they will probably be used in the future.

Mold Preparation.—Wax patterns are prepared as illustrated in Fig. 1. They are then dip-coated and invested according to the formulae shown in Table 1. These formulae were obtained through a confidential source and it has been requested that this information be kept confidential.

Some heats have been poured in which the iron oxide was omitted from the dip-coat preparation. The evidence is inconclusive, but it is thought that somewhat better surfaces are obtained if the oxide is omitted. This may be because of the fact that the iron oxide is fluxed by some of the alloys poured.

After investing, the molds are allowed to set for at least four hours after which the excess investment is removed and the molds are marked for identification. They are then dewaxed in a core oven at 275°F for two hours. The final step consists of placing them in a furnace at 1600°F where they are fired for at least 16 hours (max. 96 hours) before being poured.

Melting and Pouring.—The furnaces constructed for this project are illustrated in Figs. 2 and 3. They are small induction units lined with a stabilized zirconia crucible.

Melting stock for all heats has consisted either of purchased shot for the standard alloys or high-purity electrolytic raw materials for the heats poured in the alloy development program.

As soon as the heats are melted, a platinum-10% rhodium-platinum thermocouple encased in a fused silica protection tube is inserted into the center of the bath and the power turned off. The solidus temperature can be observed on the cooling curve which is recorded in this manner. Frequently, the thermal arrest at the liquidus temperature is missed, but it can usually be noticed upon remelting the heat under very low power input. The heat is then superheated under low power input (low power so that the heating rate will be slow enough to control) until the desired pouring temperature of 250°F in excess of the liquidus temperature is reached. This pouring temperature was decided on, on the basis of a series of heats poured in Haynes Stellite 31 (previously reported).

Throughout the course of the melting operation, argon gas is flushed over the surface of the melt to minimize oxide formation. A refractory brick (with a small hole for the thermocouple) is placed on top of the furnace to assist in keeping an argon atmosphere over the melt.

Immediately prior to pouring the heat, the hot (1600°F) mold is clamped on the furnace as shown in Fig. 3. Then the furnace is rotated and the argon flow is increased until a line pressure of approximately 5 psi occurs. It was shown in an earlier report how this pouring under increased pressure increased the fluidity of the metal and permitted lower pouring temperatures.

### DISCUSSION OF RESULTS

Standard Materials.—At the time of initiation of this project it was agreed that the first objective (alloy development) should be preceded by proving that the technique used at the University Cast-Metals Laboratory produced results which were at least comparable to those reported in the literature for standard materials such as HS 31.

The test results on these standard materials, made according to the procedure just described, are given in Table II. Some of these data have been previously reported, and some have not. Heat R83, for example, is a control heat which was poured during the alloy program to check that the procedure evolved in earlier stages was still satisfactory. Although the elongation on one bar of this heat was slightly under the specified minimum, in general the properties were satisfactory enough to indicate that the process has remained in control. Also included in the table for general information are some data on a heat of GMR 235 which was made according to the same procedure on another project at the University.

In addition, some data on shell molds are included. These results will be discussed in a subsequent section but are included in Table II for ease of comparison.

It is thought that these data illustrate that the technique evolved is at least comparable to what others are doing since the end results are similar or superior. A further illustration of the reproducibility of the procedure is shown by duplicate results obtained on special heats in the alloy program. (Table III - Heats 81 vs 82, Heat 94 vs 101, Heat 91 vs 106.)

Alloying Program.—Most super alloys in use today are complex mixtures of from five to ten elements. The precise role of most of these elements and their interrelation is not known completely, especially in cast alloys. In an attempt to answer some of these questions and in order to understand the microstructures of heat-resistant alloys, the decision was made to make a series of heats in which only one alloying element was added at a time. By studying the properties and structure of these heats, it is hoped that a more fundamental understanding of the mechanism of strengthening and substitutional elements may be gained. The eventual goal, of course, is to develop an alloy, low in strategic materials, which will have properties as set forth in the introduction to this report.

The base analysis chosen for this work is a ternary alloy consisting of 18% nickel (enough to form stable austenite) with 18% chromium (for oxidation resistance), and the balance iron. To this base analysis various amounts of other elements were added. The stress-rupture properties of some of these heats are given in Table III and some of the most interesting microstructures

are shown in Figs. 4 through 9. The information which has been obtained to date will be discussed under each specific element added to the FeNiCr base.

Base Alloy: 18% Ni-18% Cr.—No photomicrograph of this material has been included because of the difficulty of developing grain boundaries in this single-phase material. The structure apparently is a homogeneous austenite (as was expected), usually with some inclusions which are probably oxides. A dendritic pattern has been observed with some of the etches.

Considerable difficulty was encountered in producing clean, sound heats of this simple ternary alloy. Part of the difficulty was finally traced (it is believed) to a batch of electrolytic nickel which had what seemed to be an abnormally high hydrogen content. Or it may be that this alloy, consisting of only iron, nickel, and chromium, is very susceptible to gas pickup. In all probability, the difficulty in producing heats is expressed in the rather poor reproducibility of the 1500°F tests.

Aluminum Heats: 5% Al - Fig. 4.—Aluminum is a ferrite former. The addition of 5% Al, combined with 18% Cr, which is also a ferrite former, was sufficient to cause the formation of some ferrite in the microstructure. At 2% Al a very small quantity of ferrite was formed, and at the 1% level practically no ferrite was formed. These microstructural observations were confirmed by a rough magnetic index. (A magnet was suspended by a twelve foot piece of twine. The distance which a specimen pulled the magnet is a rough measure of magnetism.)

At the 5% level the 1500°F tensile strength of the base was increased from about 16,600 psi to 20,400 psi and the 100-hour rupture strength from an average of 4500 psi to 7500 psi.

Titanium Additions: 5% Ti - Fig. 5.—According to the iron-titanium phase diagram, titanium also is a gamma-loop closer, i.e., a ferrite stabilizer. The precipitate shown in Fig. 5 is not thought to be ferrite, however, because the specimens had practically none of the magnetism which should have been present if the precipitated phase were ferrite. The structure shown in Fig. 5 is obviously not an equilibrium structure as evidenced by the fact that the center of the precipitated phase was preferentially attacked by the etchant. Further work is needed to identify this phase.

The addition of 5% titanium increased the hot tensile strength of the basic alloy quite markedly. Unfortunately, most of the bars cast in this heat were of inferior quality so that a rupture life could not be established.

<u>Carbon Series: 0.5% C - Fig. 6.— The data on the carbon series present somewhat of a paradox. 0.2% C raised the hardness of the base alloy and 0.5% carbon raised it still more, as would be expected. The structure of the 0.5% C alloy consists of typical primary carbides [probably (Fe-Cr) $_{23}$ C<sub>6</sub>]</u>

at the grain boundaries with secondary carbides precipitated in the matrix adjacent to the grain boundaries. The 1.0% carbon heat, however, was softer than the others, apparently less magnetic, and the strength was lower and the ductility higher than the 0.5% C heat. In addition, the microstructure appeared to have fewer carbides than the 0.5% C heat. In all probability, the heat was off analysis (this is now being checked) and a repeat heat is in order.

Three successive heats were run at the 0.5% C level for the purposes of studying dip-coat formulae (iron oxide experiments previously referred to). Hot tensile tests on two of these heats duplicated each other very well (see table).

Boron: 0.5% B - Fig. 7.—One heat has been made to determine the nature of the boron precipitate. This structure is illustrated in Fig. 7. It has not been positively identified but appears to be a eutectic. Since there is an iron-boron eutectic at 3.8% B in iron-carbon alloys, the phase shown may be related to this constituent.

The addition of .5% B resulted in a moderate increase in the hot tensile strength.

Molybedenum Series: 15% Mo - Fig. 8.—Molybedenum forms the hard, brittle, sigma phase in iron-nickel-chromium alloys. At 25% Mo, the alloy was so brittle that the test bars were broken with normal handling during cleaning operations. Fifteen percent molybedenum produced the structure shown in Fig. 8. This alloy was quite strong. Stress-rupture tests to establish the 10- and 100-hour life rupture strengths are now in progress. Some of this phase (either  $\text{Fe}_3\text{Mo}_2$  or sigma) was also noticed in the 5%-molybedenum heats.

Because of the potent strengthening effects of molybedenum, and its relative availability, this alloying element will probably play an important role in subsequent phases of the alloy development program.

<u>Cobalt</u>: 25% <u>Co</u> - <u>Fig</u>. 9.—For completeness, and in spite of the strategic importance of cobalt, it was thought necessary to acquire some basic data on cobalt additions. For some unexplainable reason, a good deal of trouble has been encountered in making a good 25%-cobalt heat. The material seems to be unusually susceptible to dirt.

Only a moderate improvement in hot tensile strength was found. Probably, however, the longer-time rupture strengths would be raised more than is indicated by the hot tensile strength. The structure is essentially austenite. The grain size of this alloy is much smaller than the base alloy appears to be, but since a good etchant for the base material has not been found, this tentative conclusion will have to await confirmation.

Manganese Substitution for Nickel.—One heat has been made in which 18% manganese was substituted for 18% nickel. A tremendous amount of scum was formed on the melt and the surface quality of the bars was very poor. The alloy was strongly magnetic, indicating a substantial amount of ferrite in the microstructure. Since a large quantity of manganese may have been oxidized (no chemical analyses are available yet), the material may be more ferritic than if it had actually contained 18% manganese. Potentially, manganese is an attractive substitute for nickel; a considerable amount of future work is planned with this element.

### PART II. CASTING PROCESSES

The lost-wax investment-casting process is a long, laborious and expensive way to form metal into the desired shape. A very large percentage of the cost of investment castings is due to the method, not the alloy which is cast. Of the other methods of producing precision castings, the Croning shell-molding process was considered the most attractive substitute for a number of reasons.

Consequently, a metal test-bar pattern for shell molding was prepared (Fig. 10) and experiments performed to determine the surface quality and dimensional tolerances attainable and to see if the stress-rupture properties of standard alloys made by this process were the same as in the investment-casting process. These three factors, surface, dimensions, and properties, will be discussed separately.

#### SURFACE

The surface quality of any casting is a function of the surface of the mold (provided there is no mold reaction) and the pouring technique (especially fluidity). If the mold surface is rough, the casting will be rough. The principal reason why investment castings have good surface quality is that the smooth wax or plaster patterns are coated with a fine dip-coat against which the metal freezes. There is no fundamental reason why an equally fine surface could not be obtained by the shell-molding process.

Although comparable surfaces have not been produced to date, the problem is not thought to be insurmountable. For example, Fig. 11 shows one-half of a shell mold where the quality of the surface varies with the degree of packing of the sand, which, in turn, is a function of the method of making the shells. The surface of the casting, Fig. 12, faithfully reproduced the surface quality of the mold. Where the shell was rough, the casting was rough, and vice versa (the casting is a mirror image of the shell in the

photograph). For this reason, it is hoped that the surface roughness can be controlled within desired limits.

#### DIMENSIONS

In both the investment casting and shell processes, the mold is made from a metal pattern, directly in the case of the shell mold, and indirectly in the care of investment molds. Fundamentally therefore, there is no reason to expect that one process should give closer dimensional tolerances than the other, provided the surface quality is the same in both cases. In fact, actual micrometer measurements of test bars made from both molding methods show the same degree of tolerance (+ .001 in. to .002 in. on a .250 in. diameter specimen). Both castings are usually thicker across the parting line than along the parting. Part III of this report outlines some work planned for the future along this line.

#### PROPERTIES

It was found that shell-molded castings could be safely poured at a somewhat lower temperature than investment molds. And, significantly, much less trouble has been encountered in producing clean, sound, test bars in shell molds. The stress rupture properties of a few heats of shell-molded HS31 and HS21 bars are listed in Table 2. The properties approach those of the investment cast bars in most cases. Since the surface quality of these bars was admittedly not equal to the investment cast bars, and since a rough surface frequently impairs the properties of these types of materials, the slightly inferior results are not considered to be the fault of the shell molding process per se. Thus there seems to be no metallurgical reason why shell molded heat-resistant alloy castings would not be satisfactory.

### PART III. FUTURE WORK

#### ALLOY PROGRAM

The development of a suitable alloy has just begun. In addition to those heats listed in Table III, a few more heats in the preliminary program are planned:

1. Additional heats in series already started where promising results are indicated or where additional data are needed (e.g., the carbon series is perplexing).

- 2. Further work on substitution of manganese for nickel.
- 3. A nitrogen-nickel-chromium and a nitrogen-manganese-chromium series.
- 4. Study of vacuum melting, vacuum premelting, air melting in comparison with argon melting of a standard alloy, and perhaps on one or two other promising alloys.

Some work still needs to be done on the heats already poured (and those listed above) to understand the structure and microstructure. X-ray diffraction and/or electron micrographs may be needed. In addition, quantitative room-temperature tensile data are still needed on the alloys already poured.

When these heats are evaluated sufficiently enough to make some intelligent guesses, the actual task of trying to put together the correct amounts of several elements to obtain the properties required, with a minimum amount of strategic (or prohibitively expensive) alloys will be started. It is estimated that this phase of the work can commence early in the fall.

#### CASTING PROCESSES

The sponsors biggest concern with the shell-molding process is the dimensional (and surface) problem. In an effort to answer some of these questions in a more quantitative way, a prototype pattern will be constructed shortly and experiments run. The shell-molding equipment is being modified in an attempt to produce more uniform surfaces, and still other experiments with refractory mixtures are planned to make a finer surface.

Another possibility for producing the turbine rotors is that of making an investment core to be inserted into a shell mold. Such an investment core would not be nearly as expensive as a whole casting made by the investment process and it would be more amenable to mass production.

#### TABLE I

### SUMMARY OF INVESTMENT CASTING PROCESS

#### Investment:

### Refractory materials:

12.0% 200 mesh silica flour 23.5% 40-80 mesh silica sand 25.0% G. Grog 40.0% P. Grog

.4% MgO (powdered)

Dry ingredients mixed thoroughly in cement mixer for 30 minutes.

### Investment Binder:

2996 cc Ethyl Silicate 40 2385 cc Diluter(5% H<sub>2</sub>O, 95% Synasol-ethyl alcohol) 619 cc Reactor (50% H<sub>2</sub>O, 50% Synasol + .75% HCl)

Diluter mixed with ethyl silicate and then reactor added. When these are mixed the liquid binder is added to the refractory and blended in the cement mixer.

## Dip-coat: (10 lb mix)

### Dry materials:

10 lb 200 mesh silica flour
.25 lb Fe0
13.5 g sodium fluoride
Dry materials blended thoroughly

### Wet materials:

875 cc Nalcoag (National Aluminate Co., Chicago) 675 cc H<sub>2</sub>O

Wet materials mixed and added to dry materials. Ten minutes prior to using 60 cc of batch A (below) and a few drops of octyl alcohol (defoaming agent) are added.

Batch A - wetting agent 40 g Wetnal 1000 cc H<sub>2</sub>O

After patterns are dipped they are sprinkled while wet with 40 mesh silica and allowed to dry for four hours prior to investing.

			TA	TABLE 2			
		PROPERTIES OF	GS OF STANDARD	HIGH TEM	HIGH TEMPERATURE ALLOYS	OYS	
Molding Method	Heat No.	Type Material	Test Temperature °F	Stress	Life, hrs	Elongation in one inch	Reduction of Area
Investment Cast	R8	HS31	1500°F	30000	106.9	12.1	15.4 21.4
Investment Cast	R83	HS51	1500°F	30000	21.1 31.9	0.00	8.5
Shell Mold	R102	HS51	1500°F	30000	40.1 37.3	000	3.0 5.0 (1)
	Spec.	HS31	1500°F	30000	15.0	0.9	1 0, 2
Investment Cast	R18	HS21	1500°F	20000	46.8	4.9	4.9
Shell Mold	R72	HS21	1500°F	30000	5.7	6.0 7.0	8.0 11.0 (1)
Shell Mold	R73	HS21	1500°F	30000	4°L 8°6	86.5	12.5 8.0 (1)
	Spec.	HS2.1	No specification within range	ıs. of	All life fi	figures shown above values.	ove are
Investment Cast	R43	GMR235	1500°F	35000 35000 35000	397. 317. 384.	9.0 10.0 13.0	15.0 15.0
	Typical values (2)	GMR235	1500°F	35000	260 (2) 290	6 (2) 11	i i
(1) Nonstandard	tensile specim	cimen - 1/2"	"g.l.x.250"	"diam.	(2) General Motors Trans. AFS, Vo	paper 1 62, 1	published in 1954, p. 295.

TABLE 3 PROPERTIES OF HEATS POURED ON ALLOY PROGRAM

	Naumal Campanthi			As Cast Physical Properties			Properties at 1500°F				1500°F Summary Properties Hot 10 hr 100 hr		
Heat No.	Norma	1 Composit	ion	Rockwell Hardness	Magnetic Index (1)	Reference Fig. No.	Stress psi	Life Hours	Elongation %	Reduction of Area,%	Hot Tensile Strength	10 hr Rupture Strength	Ruptur
Base Alloy													
R26	18% Ni	18 <b>%</b> Cr		B36-44	1/8"		15,150 6,000 3,000	STTT (2) 1.5 163	10.0 20.0 Broke in	11.0 22.0			
R37	18% Ni	18% Cr		B42-45	1/4"		14,500	STTT 1.8			14,500	4.500	3,200
R40	18% Ni	18% Cr		B29-42	1/4"		7,000 18,750 7,000 5,000	STTT 10.7 262.0	24.0 7.0 5.0	42.0 } 6.0	18,750	4,500 7,800	3,200 5,600
R45	18 <b>%</b> Ni	18 <b>%</b> Cr		B41-49	1/8"		18,000 7,000 5,000	STTT 19.0 107	12.0 12.0 12.0	19.0 10.5			
Aluminum Heats										-			
R∱1	18% Ni	18 <b>%</b> Cr	5% Al	в63-67	16"	Fig. 4	20,400 10,000 8,000 7,000	STTT 10.8 86.5 171	46.5 23.0 17.0 18.0	55.5 43.0 22.0 28.5	20,400	10,100	7,500
R44 R62 R63	18% Ni 18% Ni 18% Ni	18% Cr 18% Cr 18% Cr	2% Al 2% Al 1% Al	B36-38 B38-39 B46-47	3/8" 3/8" 5/8"		7,000	111	16.5	23.5			
<u>Titanium Heats</u> R93 R66	18% Ni 18% Ni	18% Cr 18% Cr	5% Ti 2% Ti	B71-73 B17-35	1/8" 3/8"	Fig. 5	34,200	STTT	14.0	27.5	34,200		
Carbon Heats R48	18 <b>%</b> Ni	18% Cr	1 <b>%</b> C	B52 <b>-</b> 65	1/8"		19,400 10,000 6,500 5,500	STTT 6.3 69.6 137	23.0 19.0 10.0 8.0	25.5	19,400	9,100	5,90
R65 R80 R82 R81	18% Ni 18% Ni 18% Ni 18% Ni	18% Cr 18% Cr 18% Cr 18% Cr	.5% C .5% C .5% C .5% C	B80-83 B79-75 B71-76 B73-80	1/4" 1/4" 1/2" 1/8"	Fig. 6	30,600 30,100 17,000 14,000 11,500	STTT STTT 23.7 45.0 108.0	13.0 10.0 3.0 4.5 Broke in	27.0 26.0 5.5 5.5 fillet	30,100 30,600		11,60
R64	18% Ni	18% Cr	.2% C	R63-64	1/4"		8,500	In Fest					
Boron Heats R105	18% Ni	18 <b>%</b> Cr	.5% B	B64-66	1/8"	Fig. 7	23,200 10,000	STTT 60.1	34.0 41.0	47.5	23,200		
Molybedenum Heats													
R113 R90 R94	18% Ni 18% Ni 18% Ni	18% Cr 18% Cr 18% Cr	25% Mo 15% Mo 15% Mo	C37-49 C24-28 C18-24	1/8" 1/8" 1/16"	Fig. 8	Ver 60,200	y brittle STTT	- Bars fractu	red at shake	out.		
R101	18% Ni	18% Cr	15% Mo	C16-25	1/4"	rig. O	55 <b>,</b> 800+	STTT	Broke in	threads	55,800+ 60,200		
R68	18% Ni	18 <b>%</b> Cr	5% Mo	B48-59	1/4"		30,000 23,500	2.8 STTT	26.0 15.0	36.0 42.0	23,500		
Cobalt Heats										,			
R91 R104 R106 R112 R67 R74	18% Ni 18% Ni 18% Ni 18% Ni 18% Ni 18% Ni	18% Cr 18% Cr 18% Cr 18% Cr 18% Cr 18% Cr	25% Co 25% Co 25% Co 25% Co 5% Co	B38-45 B46-47 B48-58 B43-51 B28-41 B39-44	1/4" 5/8" 3/8" 3/8" 1/4" 3/8"	Fig. 9	22,400 25,500	STTT STTT	26.0 17.0	23.0	22,400 25,500		
Manganese Substitution for													
R107	0% Ni	18% Cr	18% Mn	в67-68	40"		a a v		Now in progre				

<sup>(1)</sup> Magnetic index - An Alnico magnet was suspended from a 12' string. The amount of displacement by a tensile bar is a measure of the magnetic force.
(2) STTT means short time tensile test.

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