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HIGH TEMPERATURE ALLOYS

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This is a continuation-in-part of application Serial No. 119,902, filed June 27, 1961, now Patent No. 3,165,400.

This invention relates to austenitic alloys for high temperature applications of the general character disclosed and claimed in the copending application of Amedee Roy, one of the present applicants, and Walter E. Jominy, Serial No. 119,902, filed June 27, 1961, which by this cross reference is made a part hereof, and especially to such composition made to include or exclude trace amounts of certain elements in order to improve the elevated temperature properties of the compositions and/or minimize variations in the properties between heats of apparently the same composition.

Early work with alloys of the above type had often shown a wide range in properties between heats of apparently the same composition (insofar as the elements thereof could be readily analyzed by conventional techniques). It was theorized that trace elements picked up either from the raw materials or the melting crucible were strongly influencing elevated temperature properties and preventing consistent properties from being attained. Their effect often was to lower the property values expected of the alloy in the absence of these effects.

Extensive investigations were conducted to investigate the effects of various beneficial or detrimental trace elements that could possibly be present in the alloy. To this end small melts (approximately 3600 gms.) were prepared of the purest raw materials (commercial grades) available plus small additions of one of the following elements: B, Zr, Ti, Ta, P, S, and Al. For example, six heats of pure materials were prepared with Zr additions ranging from 0.005 to 1% by weight and similar heats were prepared with the others. In some cases two of the trace elements, for instance Zr and B were added together to determine if their effects were complimentary or additive. Moreover similar melts were prepared in four different types of melting crucibles: Al_2O_3, MgO, Zircon ($Zr_2O_3 \cdot SIO_2$) and graphite. The small heats were cast into investment molds (both solid and shell type) containing approximately ten test bar cavities (¼" dia. gage section). These cast to size test bars were utilized to determine the elevated temperature properties of the heats.

Table I shows the composition of the alloys tested and the amounts of trace elements included. The amount of iron (Fe) is not shown, it constituting essentially the remainder thereof.

Table II shows the stress rupture life under various loads and the stress for a rupture life of 100 hours, at

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the indicated temperature to provide a convenient basis for comparison.

The composition of a typical heat before adding any of the trace elements may be seen from heat No. 267D.

With respect to zirconium (Zr) it will be noted from heats 293D, 272D, 273D, 277D, 320D, and 321D in Tables I and II for example, representing compositions containing respectively 0.005, 0.01, 0.1, 0.25, 0.5 and 1.0 weight percent zirconium added to the base composition 267D that best results were obtained with heat 273D containing 0.1 zirconium. It was noted too that single additions of zirconium up to 1.0% did not substantially adversely affect the basic composition property. In Table II the letter A appearing after the heat number means that the as-cast alloy was solution heat treated at 2250° F. for ½ hour and aged at 1400° F. for 20 hours after air cooling.

With respect to boron (B), it will be noted from heats 292D, 285D, 268D, 286D and 269D in Tables I and II, for example, representing compositions containing respectively 0.002, 0.005, 0.01, 0.025 and 0.05 weight percent boron added to the base composition 267D that the optimum properties in the A (solution treated and aged) condition as shown in Table II was obtained with heat 285D containing 0.005 boron. The properties of heats 286D and 269D containing 0.025 and 0.05 boron were adversely affected by these amounts of boron. It would appear that up to about 0.025% boron may be safely used in the base composition 267D preferably up to about 0.01%. For the same heats in as cast condition those containing 0.025% and above were inferior to those containing less than this amount of boron. The use of boron is especially desirable because it appears to promote an optimum distribution of the precipitates and an optimum structure . With respect to a combination of boron and zirconium heats 274D, 275D, 278D and 279D it appeared that the combination in the optimum amount used separately did not produce any improved properties in the base alloy containing single additions of these elements.

With respect to trace elements phosphorous, sulfur, titanium, tantalum and aluminum as reported by heats 290D, 291D, 322D, 323D, 299D, 301D, 295D, 296D, 297D and 324D, 388D, 389D and 391D, it appears that trace amounts of these elements are not detrimental. However, 2% or more titanium is detrimental and phosphorous additions at 0.5% and above produced alloys which did not respond to heat treatment.

With respect to the crucibles used for melting the heats it was found that part of all of the trace elements given above may be picked up therefrom. Heats 243D, 246D, 247D and 248D were, for example, prepared using respectively graphite, alumina, zircon, and zircon crucibles and picked up at least some of the trace elements therefrom. Most of the other heats were prepared in magnesia crucibles which are less likely to impart the trace elements to the heats.

Table I.—Chemical compositions of heat (nominal weight percent)

Alloy	C	Cr	Si	W	Mo	Cb	Ni	Mn	N	B	Others
243D	1.0	18.0	0.5	2.0	2.0	2.0	5.0	5.0	0.3	------	Graphite crucible.
246D	1.0	18.0	0.5	2.0	2.0	2.0	5.0	5.0	0.3	------	Alumina crucible.
247D	1.0	18.0	0.5	2.0	2.0	2.0	5.0	5.0	0.3	------	Zirconia crucible.
248D	1.0	18.0	0.5	2.0	2.0	2.0	5.0	5.0	0.3	------	Do.
263D	1.0	18.0	0.5	2.0	2.0	2.0	5.0	5.0	0.3	------	0.1 Ti.
265D	1.0	18.0	0.5	2.0	2.0	2.0	5.0	5.0	0.3	------	0.5 Ti.
267D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	MgO crucible.

Table I—Continued

Alloy	C	Cr	Si	W	Mo	Cb	Ni	Mn	N	B	Others
268D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	.01	
269D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	0.5	
272D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.01 Zr.
273D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.1 Zr.
274D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	.01	.01 Zr.
275D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	.01	.1 Zr.
277D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.25 Zr.
278D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	.01	.1 Zr.
279D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	.005	.1 Zr.
285D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	.005	
286D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	.025	
290D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.005 P.
291D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.02 P.
292D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	.002	
293D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.005 Zr.
295D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.005 Ti.
297D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.5 Ti.
299D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.01 S.
301D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.05 S.
320D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.5 Zr.
321D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	1 Zr.
322D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	0.5 P.
323D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	0.1 P.
324D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	2 Ti.
325D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	AlO_3 crucible.
326D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.01 Zr.
327D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	0.1 Zr.
328D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	0.2 Zr.
329D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	0.4 Zr.
388D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.1 Zr. .01 Al.
389D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.1 Zr. .05 Al.
390D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.1 Ti. .1 Zr.
391D	1.0	18.0	0.5	1.0	1.0	1.0	5.0	5.0	------	------	.1 Zr. .1 Ta.

Table II.—Mechanical Properties of Heats

Alloy	Stress-rupture data at 1,500° F.			
	Stress	Time	Elong., percent	100 hr. life
243D	27,000	39.4	20	23,000
243D	25,000	58.7	20	
243D	22,500	109.9	20	
246D	27,000	49.6	13	24,500
246D	25,000	93.3	23	
246D	22,500	212.3	25	
247D	27,500	46.1	24	24,500
247D	25,000	76.6	12	
247D	22,500	199.2	15	
248D	27,000	44.4	20	24,000
248D	25,000	40.7	14	
248D	22,500	94.3	14	
248D	22,500	219.0	15	
248D	20,000	453.3	16	
263D	27,000	65.8	19	26,000
263D	27,000	93.4	20	
263D	25,000	162.5	17	
263D	22,000	343.5	22	
265D	27,000	51.8	13	23,500
265D	25,000	78.4	20	
265D	22,500	126.3	24	
267D	27,000	58.9	------	25,000
267D	25,000	88.6	19	
267D	22,500	271.6	13	
267DA	30,000	101.6	------	27,500
267DA	30,000	28.6	6	
267DA	27,000	54.0	5	
267DA	27,000	35.7	10	
267DA	25,000	238.4	5	
268D	30,000	8.4	24	25,500
268D	27,000	49.5	20	
268D	25,000	130.5	11	
268DA	32,000	22.2	11	29,500
268DA	30,000	112.2	6	
268DA	27,000	286.7	5	
269D	30,000	9.5	23	23,500
269D	27,000	25.0	16	
269D	25,000	57.6	24	
269D	22,500	233.3	19	
269DA	27,000	18.1	1.5	24,500
269DA	27,000	0.6	1	
269DA	25,000	82.4	3	
272D	30,000	14.0	20	24,500
272D	27,000	39.6	16	
272D	25,000	93.4	15	
272DA	35,000	10.9	8	31,000
272DA	32,000	66.9	3	
272DA	30,000	144.1	2	
273D	30,000	16.1	16	26,500
273D	27,000	72.4	15	
273D	25,000	162.7	12	
273DA	35,000	10.8	12	30,000
273DA	32,000	60.7	2	
273DA	30,000	103.3	3	
274D	30,000	7.2	16	25,000
274D	27,000	42.3	20	
274D	25,000	75.5	15	
274DA	32,000	23.1	2	27,000
274DA	30,000	45.0	5	
274DA	27,000	92.2	5	
275D	27,000	39.9	20	25,000
275D	30,000	13.9	17	
275D	25,000	121.8	16	
275DA	35,000	16.3	5	29,000
275DA	32,000	30.6	6	
275DA	30,000	82.7	4	
277D	30,000	13.5	17	25,500
277D	27,000	44.9	19	
277D	25,000	190.3	7	
277DA	32,000	34.7	6.5	29,000
277DA	30,000	68.0	4	
277DA	27,000	238.8	2.5	
278D	27,000	31.6	20	25,000
278DA	32,000	32.1	7	30,000
279D	27,000	31.6	20	25,200
279D	30,000	5.8	19	
279D	27,000	29.6	15	
279D	25,000	126.0	14	
279DA	32,000	13.4	5	27,500
279DA	30,000	34.3	5	
285D	30,000	6.9	23	25,500
285D	27,000	37.0	15	
285D	25,000	139.9	12	
285D	30,000	10.3	19	
285DA	35,000	1.6	1	30,000
285DA	32,000	43.5	6	
285DA	30,000	140.6	3.5	
286D	30,000	6.9	23	23,500
286D	27,000	14.4	23	
286D	25,000	54.0	16.5	
286DA	30,000	22.1	1	26,500
286DA	27,000	0	1	
286DA	27,000	6.8	1	
290D	30,000	9.7	17	26,200
290D	27,000	55.3	14	
290D	25,000	215.1	10	

Table II—Continued

Alloy	Stress-rupture data at 1,500° F.			
	Stress	Time	Elong., percent	100 hr. life
290DA	32,000	53.7	4.5	29,000
290DA	30,000	47.1	7	
290DA	30,000	5.0	1.5	
290DA	27,000	0.2	1	
290DA	27,000	9.9	2	
291D	30,000	14.2	17	25,200
291D	27,000	33.0	22	
291D	25,000	139.5	15	
291DA	35,000	2.5	1	31,000
291DA	35,000	2.3	2	
291DA	32,000	103.9	5	
291DA	30,000	102.5	7	
292D	32,000	2.6	26	26,000
292D	30,000	11.0	27	
292D	27,000	66.8	18	
292D	25,000	124.7	16	
292DA	32,000	5.8	1	27,000
292DA	30,000	21.9	0	
292DA	27,000	75.6	2	
293D	32,000	2.5	22	26,000
293D	27,000	66.4	15	
293D	25,000	140.1	14	
293DA	32,000	10.4	2	28,500
293DA	30,000	42.7	1	
293DA	30,000	94.7	1	
293DA	27,000	157.7	2	
295D	30,000	10.0	28	26,000
295D	27,000	52.3	19	
295D	25,000	126.8	23	
295DA	35,000	7.3	14	31,000
295DA	32,000	77.7	5	
295DA	30,000	119.4	5	
296D	30,000	8.7	26	25,500
296D	27,000	72.0	18	
296D	25,000	107.8	26	
296DA	35,000	11.1	8	30,000
296DA	32,000	59.6	2	
296DA	30,000	73.2	3	
296DA	30,000	74.2	3	
297D	30,000	5.2	13	25,500
297D	27,000	62.8	15	
297D	25,000	107.8	15	
297DA	35,000	18.1	5	30,500
297DA	32,000	70.4	3	
297DA	30,000	119.3	3	
299D	30,000	9.4	21	24,500
299D	27,000	36.7	17	
299D	25,000	64.6	15	
299DA	35,000	22.6	7	30,500
299DA	32,000	61.1	3.5	
299DA	30,000	108.8	3	
301D	30,000	6.6	19	25,500
301D	27,000	37.2	15	
301D	25,000	119.4	12	
301D	25,000	133.8	12	
301DA	35,000	25.7	7	30,000
301DA	32,000	49.2	7	
301DA	30,000	137.5	4	
320D	30,000	24.7	14	25,500
320D	27,000	41.7	----------	
320D	25,000	165.5	10	
320DA	32,000	23.9	4	28,000
320DA	30,000	43.5	3	
321D	30,000	8.4	19	25,800p
321D	27,000	56.8	16	
321D	25,000	146.1	13	
321DA	35,000	10.2	3	28,500
321DA	32,000	33.3	2.5	
321DA	30,000	49.7	2	
322D	30,000	41.6	10	27,500
322D	27,000	96.6	5	
322D	25,000	233.5	5	
322DA	30,000	12.3	2	24,000
322DA	27,000	4.5	0	
322DA	27,000	4.5	0	
323D	30,000	48.0	10	27,000
323D	27,000	87.5	11	
323D	25,000	204.2	8	
323DA	30,000	25.9	1	24,500
323DA	27,000	51.9	1	
323DA	25,000	82.1	1	
324D	20,000	73.8	7	19,500
324D	25,000	0.6	2	
324DA	25,000	82.1	1	24,500
324DA	25,000	0.5	6	
324DA	22,500	0.4	4	
324DA	20,000	0.9	----------	
325D	30,000	19.6	19	26,500
325D	27,000	79.8	16	
325D	25,000	241.6	10	
325DA	35,000	53.0	3	32,500
325DA	32,000	98.8	3	
325DA	30,000	93.1	4	
325DA	30,000	192.0	2	
326D	30,000	11.4	20	26,500
326D	25,000	216.0	9	
326D	27,000	89.6	13	
326DA	35,000	30.7	4	31,500
326DA	32,000	63.8	3	
326DA	30,000	165.2	2	
327D	30,000	31.1	16	27,500
327D	27,000	136.3	14	
327D	25,000	256.4	----------	
327DA	35,000	28.3	5	31,500
327DA	32,000	87.2	4	
327DA	30,000	174.8	3	
328D	30,000	16.3	18	26,500
328D	27,000	69.8	17	
328D	25,000	271.0	7	
328DA	32,000	2.2	1	29,000
328DA	30,000	65.0	3	
328DA	30,000	69.7	2	
329D	30,000	25.7	16	26,000
329D	27,000	73.1	12	
329D	25,000	152.9	8	
329DA	35,000	2.2	1	29,000
329DA	32,000	22.6	3	
329DA	32,000	15.4	3	
329DA	30,000	61.5	2.5	
388D	27,000	53.6	13	26,000
388D	30,000	12.3	19	
388D	25,000	188.1	9	
388DA	30,000	105.2	3	30,500
388DA	32,000	45.5	3	
388DA	27,000	332.2	2	
389D	25,000	83.4	10	26,500
389D	27,000	113.3	8	
389D	30,000	4.7	14	
389D	25,000	153.1	10	
389DA	30,000	59.5	3	29,000
389DA	27,000	218.9	3	
390D	25,000	97.7	14	24,500
390D	27,000	18.7	16	
390D	22,500	402.3	9	
390DA	30,000	76.4	3	29,000
390DA	27,000	154.9	2	
391D	25,000	208.6	8	25,800
391D	27,000	37.8	13	
391DA	30,000	85.4	3	29,500
391DA	27,000	238.3	2	

We claim:

1. An iron base casting type alloy for producing products of complex shape and having substantial oxidation resistance and high strength at elevated temperature and characterized by a structure having a network outlining an austenitic phase embedding fine dot-like precipitates, said alloy consisting essentially of by weight percent, about 0.6 to 1.6% carbon; about 12 to 35% chromium; between 0 to 2.5% silicon; between 0.2 to 12% of a plurality of carbide forming elements from the group consisting of tungsten in the range 0.1 to 10%, molybdenum in the range 0.1 to 9%, columbium and tantalum, said columbium and tantalum combined being in the range 0.1 to 5%, up to 15% of metal from the group consisting of, nickel in the range 0 to 15%, manganese in the range 0 to 15%, cobalt in the range 0 to 8%; 0 to 0.6% nitrogen; a small but effective amount of boron not exceeding 0.025% to promote distribution of the precipitates; the balance essentially iron, said iron content being at least 40%, said nickel being at least about 2% when said manganese is less than about 1%, said manganese being at least between 2 to 10% when the nickel is less than 2% and nitrogen is present, at least between about 5 to 10% when substantially no nitrogen is present and the nickel is less than about 2%, and at least about 10% when the nickel is zero.

2. An iron base alloy as claimed in claim 1 wherein

a test bar thereof ¼" in diameter exposed to 1500° F. for 100 hours is generally characterized by a structure having an interdendritic network of substantially lederburite phases outlining an austenitic phase embedding relatively fine dot-like precipitates randomly distributed therein.

3. An iron base casting type alloy for producing products of complex shape and having substantial oxidation resistance and high strength at elevated temperature, said alloy consisting essentially of by weight percent, about 0.6 to 1.6% carbon; about 12 to 35% chromium; between 0 to 2.5% silicon; between 0.2 to 12% of a plurality of carbide forming elements from the group consisting of tungsten in the range 0.1 to 10%, molybdenum in the range 0.1 to 9%, columbium and tantalum, said columbium and tantalum combined being in the range 0.1 to 5%, up to 15% of metal from the group consisting of, nickel in the range 0 to 15%, manganese in the range 0 to 15%, cobalt in the range 0 to 8%; 0 to 0.6% nitrogen; a small but effective amount of zirconium not exceeding 1.0% to promote the 100 hour stress rupture strength at 1500° F. of the alloy when heat treated; the balance essentially iron, said iron content being at least 40%, said nickel being at least about 2% when said manganese is less than about 1%, said manganese being at least between 2 to 10% when the nickel is less than 2% and nitrogen is present, at least between about 5 to 10% when substantially no nitrogen is present and the nickel is less than about 2%, and at least about 10% when the nickel is zero.

4. An iron base casting type alloy for producing products of complex shape and having substantial oxidation resistance and high strength at elevated temperature, said alloy consisting essentially of, by weight percent, about 0.6 to 1.6% carbon; about 12 to 35% chromium; between 0 to 2.5% silicon; between 0.2 to 12% of a plurality of carbide forming elements from the group consisting of tungsten in the range 0.1 to 10%, molybdenum in the range 0.1 to 9%, columbium and tantalum, said columbium and tantalum combined being in the range 0.1 to 5%, up to 15% of metal from the group consisting of, nickel in the range 0 to 15%, manganese in the range 0 to 15%, cobalt in the range 0 to 8%; 0 to 0.6% nitrogen; about 0.001 to 0.025% boron; about 0.005 to 0.1% zirconium; the balance essentially iron, said iron content being at least 40%, said nickel being at least about 2% when said manganese is less than about 1%, said manganese being at least between 2 to 10% when the nickel is less than 2% and nitrogen is present, at least between about 5 to 10% when substantially no nitrogen is present and the nickel is less than about 2%, and at least about 10% when the nickel is zero.

5. An iron base casting type alloy for producing products of complex shape and having substantial oxidation resistance and high strength at elevated temperature, said

alloy consisting essentially of, by weight percent, about 0.6 to 1.6% carbon; about 12 to 35% chromium; between 0 to 2.5% silicon; between 0.2 to 12% of a plurality of carbide forming elements from the group consisting of tungsten in the range 0.1 to 10%, molybdenum in the range 0.1 to 9%, columbium and tantalum, said columbium and tantalum combined being in the range 0.1 to 5%, up to 15% of metal from the group consisting of, nickel in the range 0 to 15%, manganese in the range 0 to 15%, cobalt in the range 0 to 8%; 0 to 0.6% nitrogen; about 0.001 to 0.025% boron; up to 2% titanium; up to 0.5 phosphorous; the balance essentially iron, said iron content being at least 40%, said nickel being at least about 2% when said manganese is less than about 1%, said manganese being at least between 2 to 10% when the nickel is less than 2% and nitrogen is present, at least between about 5 to 10% when substantially no nitrogen is present and the nickel is less than about 2%, and at least about 10% when the nickel is zero.

6. An iron base alloy as claimed in claim 3 wherein a test bar thereof ¼" in diameter exposed to 1500° F. for 100 hours is generally characterized by a structure having an interdendritic network of substantially lederburite phases outlining an austenitic phase embedding relatively fine dot-like precipitates randomly distributed therein.

7. An iron base casting type alloy for producing products of complex shape and having substantial oxidation resistance and high strength at elevated temperature, said alloy consisting essentially of, by weight percent, about 0.6 to 1.6% carbon; about 12 to 35% chromium, between 0 to 2.5% silicon; between 0.2 to 12% of a plurality of carbide forming elements from the group consisting of tungsten in the range 0.1 to 10%, molybdenum in the range 0.1 to 9%, columbium and tantalum, said columbium and tantalum combined being in the range 0.1 to 5%, up to 15% of metal from the group consisting of, nickel in the range 0 to 15%, manganese in the range 0 to 15%, cobalt in the range 0 to 8%, 0 to 0.6% nitrogen; about 0.001 to 0.01% boron; the balance essentially iron, said iron content being at least 40%, said nickel being at least about 2% when said manganese is less than about 1%, said manganese being at least between 2 to 10% when the nickel is less than 2% and nitrogen is present, at least between about 5 to 10% when substantially no nitrogen is present and the nickel is less than about 2%, and at least about 10% when the nickel is zero.

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