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CORROSION RESISTANT STEEL ARTICLE COMPRISING SILICON AND COLUMBIUM

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This application is a continuation in part of my application Serial No. 108,375 filed October 30, 1936. This invention relates to improvements in alloy steels which are resistant to heat oxidation, rust, acid and chemicals. This steel has high strength at high temperature and can be made hard and tough at atmospheric temperatures by suitable heat treatment, and is particularly adapted for valves for internal combustion engines and valve seats, turbine blades, pump shafts, boiler parts, roller bearings, chemical apparatus, tanks, bars, plates, wire and all uses where high strength and non-corrosive properties are required.

The steels of this invention are extremely resistant to the corrosive gases of internal combustion engines which are powered by high octane rating gasoline containing various quantities and forms of lead compounds, and where such corrosive lead compounds are present, the chromium content is preferably over 18%.

For those alloys of my invention which contain 18% of chromium and less, the alloys are all very resistant to high octane gasoline containing the lead compounds, but are not so resistant as those containing more than 18% chromium. Nevertheless, they give very excellent service and have the required hardness.

The chromium content of my alloy steel invention falls into three classes:

(A) From about 10% to about 18% chromium
(B) From about 18% to about 23% chromium
(C) From about 23% to about 35% chromium

In Class A the best all around efficiency is found between about 13% and 18% chromium.

In Class B the whole range seems to be efficient and does not seem to have any particular period of efficiency.

In Class C the best all around efficiency is obtained between 24% and 30% chromium.

The maximum hardness is obtained in Class A and Class C as will be more readily observed from the tables given below.

I have discovered that silicon in conjunction with columbium within my chromium and nickel range is capable of obtaining good hardness by heat treatment. Each seems to be an accentuator of the other. Neither silicon nor columbium in my range will of itself produce the necessary hardness as is more fully set out in the tables.

While it is well known that columbium can be added as an alloying element in chromium and chromium-nickel steels, columbium is always used as a softener and prevents to a very great degree inter-granular corrosion, due to carbide precipitation, when heated to the so-called sensitizing range between about 1100° F. and 1600° F.

It is well recognized that columbium and carbon form columbium carbides and chromium does not readily form carbides in the presence of columbium. The columbium is therefore inert as far as its physical effect is concerned. Under these circumstances columbium is a softening agent.

While I have no proof, it is my belief that silicon and columbium form a compound which is dissolvable in the alloy and this compound is precipitated out of the alloy to a greater or lesser degree producing hardness, probably by such precipitation; or it may even cause a phase change perhaps into one of the uncorrelated and little understood ferritic phases such as the so-called sigma phase.

I have discovered that within my chromium range of 10% to 35% chromium, silicon and columbium within my range will cause the ferrous alloy under suitable heat treatment to become hard, having a Rockwell C hardness of about 35–60, sometimes higher. It is probable that the hardness is a form of precipitation hardness (though not a usual carbide precipitate) even though regular methods of hardening are employed, such as cooling the steel in normal way from the forging temperature or heating to a high temperature in the order of 2100° F. and slowly or quickly cooling, and later reheating to a substantially lower temperature in the order of about 1200° F. to 1800° F. preferably not over 1600° F. This is a well known drawing temperature, and when it is used with chromium-nickel stainless steels it is a dangerous temperature as it causes sensitizing, meaning that the alloy is then subject to inter-granular corrosion.

All the alloys of my invention are considerably harder when cooled down from the as forged condition than when quenched from high temperature due to the fact that such cooling down from the forging temperature retains the alloy in a

strained condition and cooling down from forging temperature gives a hardness somewhat similar to the hardness effect as obtained when quenching from a high temperature plus a drawing or reheating treatment. When the alloys as forged are subjected to the drawing or reheating temperatures between 1200° F. and 1800° F. as shown in the tables, they are generally hard because of the retained strained condition or distorted crystal arrangement or probably due to the more ready precipitation of the precipitate from such a strained alloy. Although the quenching temperature shown in the tables is given as 2100° F., temperatures down as low as 1800° F. can be employed when the silicon is at the low end of the range or the carbon is high, that is, over about 1%.

I have found that within the analysis of my invention herein outlined, silicon alone within the range given, with the chromium and nickel within my range, will not produce the necessary hardness when given the heat treatment herein outlined, nor will any other form of heat treatment. Neither will columbium alone in my range and with low silicon produce the necessary hardness when given a similar type of heat treatment. But the silicon and columbium taken together within my range will produce marked hardness with excellent toughness.

The desirable hardness produced by columbium and silicon where the chromium content is less than 23% and over about 18% is not so marked as where the chromium is in excess of 24% or below 18%.

Columbium is usually added to the melt in the form of a ferro-columbium alloy. This usually contains some tantalum. My columbium range is between .5% and 5%, preferably between 1% and 3%.

The carbon content should be under 2% and preferably under 1%. For those alloys containing over 23% of chromium, the alloy will get harder with the same chromium, silicon, columbium, nickel content as the carbon is decreased. For instance, with alloys of this chromium content where the carbon is .10% or lower, the alloy is considerably harder than in the case where the same analysis is employed and the carbon is .50%. Low carbon chromium is expensive and for a good many purposes the low carbon alloy is not necessary. The use will dictate the amount of carbon that should be employed, particularly if intergranular corrosion becomes a factor, in which case the carbon content should be kept as low as feasible. Undoubtedly the medium and high carbons remove some of the effective columbium and silicon hardening characteristics by the formation of columbium carbides.

The nickel content of the alloys of my invention is between 1% and 16%. The nickel content for those alloys containing 23% or more chromium should preferably be between 2% and 16% and preferably under 10% of nickel.

Copper can be used up to about 3%; over 3% it adversely affects the forgeability of the alloy and is undesirable. It has little effect upon the physical properties of the alloy at low temperature, but does increase the hot strength of my alloy.

Manganese is a useful alloying addition giving toughness and strength at red heat temperatures. However, it causes the alloy to scale at elevated temperatures and also produces brittleness if too much is used. Under these circumstances, the manganese should not be over 4% where the chromium is over 23%; and where the alloy has less than 23% chromium, the manganese content should be graded down so that it is not more than 3%, and preferably not more than 1.5% when the chromium is 20% or lower. The manganese and nickel are not equivalents.

The silicon content should preferably be between 2% and 5% where the chromium content is less than 20%, and between 1% and 4% preferably between 2% and 3% where the chromium content is over 20% and particularly for those alloys containing more than 23% of chromium.

The elements molybdenum, tungsten, vanadium, titanium, zirconium and cobalt can be added to my alloys up to about 4% each, or an aggregate of not more than 8%. Molybdenum and tungsten in chromium-nickel steels have the well known effect of increasing the strength of this general type alloy at high temperatures and sometimes add to the toughness.

Vanadium has somewhat similar properties, but not to the same extent.

Titanium and zirconium are decided hardeners, but are intensive embrittling agents and ordinarily therefore should not be used in excess of 2%, preferably less than 1.5%. Titanium can be used as it produces hardness when used in conjunction with columbium. It will quickly induce brittleness and readily form titanium oxide in the steel, producing a dirty steel.

Cobalt has the well known property of increasing red hardness in nearly all high alloy steels, including the chromium and nickel types, and therefore can be employed in some special instances.

These high melting point elements used in conjunction with silicon alone within my range, and no columbium employed, will not produce the hardness and toughness that can be produced with the alloys within my range where the silicon and columbium are used together; therefore these high melting point elements should not be considered as substitutes for columbium and silicon, but merely as permissible additive elements and can be used for the well known properties that they convey to chromium and chromium-nickel ferrous alloys.

A general statement of permissible ranges of alloying ingredients which are embraced within my invention is as follows:

	Per cent
Chromium	10 to 35
Silicon	1 to 5
Manganese	up to 4
Nickel	1 to 16
Columbium	0.5 to 5

As stated above, the chromium content of my alloy steel invention falls into three classes, and following this division into classes, the alloys of my invention may be tabulated as follows:

Alloy ranges	Preferable range
Class A:	*Percent*
Chromium about 10.00 to 20.00%	13.00 to 18.00.
Silicon about 2.00 to 4.00%	2.00 to 400.
Nickel about 1.00 to 8.00%	4.00 to 6.00.
Manganese about 0.30 to 3.00%	.5 to 1.5.
Columbium about 0.5 to 5.00%	1.00 to 3.00.
Class B:	
Chromium about 20.00 to 23.00%	20.00 to 23.00.
Silicon about 1.5 to 4.00%	1.5 to 3.5.
Manganese about 0.3 to 4.00%	1.0 to 3.00.
Nickel about 2.0 to 10.00%	2.0 to 10.00.
Columbium about 0.5 to 5.00%	1.00 to 3.00.
Class C:	
Chromium about 23.00 to 35.00%	23.00 to 30.00.
Silicon about 1.5 to 4.00%	1.5 to 3.5.
Manganese about 0.3 to 4.00%	1.0 to 3.0.
Nickel about 2.0 to 16.00%	2.0 to 10.0.
Columbium about 0.5 to 5.0%	1.0 to 3.0.

Within the above range of alloys the carbon is preferably below 1%, although the carbon can be as low as can be made in the melt. Higher carbon can be used with high chromium of Classes B and C. Where high carbon is used with the lower chromium content, the ground mass of the alloy is robbed of its chromium by the very large amount of chromium carbides present; therefore high carbon, although producing hardness makes the alloy susceptible to corrosion and scaling. Where the chromium is over 20%, carbon as high as 2% may be employed though preferably not more than 1.5%, and those alloys containing less than 20% of chromium, not more than 1% carbon is desirable.

The alloys of my invention are sometimes initially ferritic, and at other times initially non-magnetic. I mean by initially ferritic an alloy that is magnetic, but an alloy that is initially non-magnetic does not necessarily mean that it is entirely austenitic.

With the high percentage of chromium, that is to say about 23% or over, the alloys are generally magnetic in their cast or as forged condition, or as quenched from 2100° F. or higher or lower, and when they are given the drawing temperature, that is, the reheating temperature between 1200° F. and 1600° F. up to perhaps 1800° F., but particularly between 1200° F. and 1600° F. and more particularly around about 1400° F., there seems to be produced a precipitation accompanied with hardness. This precipitation causes the alloy to become non-magnetic, or increasingly so. This is apparently not gamma iron or the formation of austenite, but apparently is a phase change, not entirely understood by me. The theoretical interpretation of such hardness is not important to the invention.

Aluminum can be used in conjunction with silicon. It increases the scale resistance, but with chromium over 23%, it is apt to produce brittleness, and for alloys containing over 23% chromium it is not a desirable addition in any sizable quantity, meaning 1% or 2%, or thereabouts. Small quantities of aluminum are often present in the alloy due to the aluminum being present in the combinations of the alloy, or arising from the degasifying of the alloy.

As previously stated, in all of the chromium-nickel alloys of this invention, silicon and columbium used separately are not hardening agents, whereas, in similar alloys where silicon and columbium are used together, considerable hardness can be produced by the reheating treatment or the release from the solid solution or solute or the hardening combination or form, whatever it may be. This is illustrated by the following table:

Heat No.	C	Mn	Cr	Ni	Cu	Si	Cb	2100° F. Water quench		1250° F. 1 hour air cooled		1400° F. 1 hour air cooled		1400° F. 15 hours air cooled		1600° F. 1 hour air cooled	
								RC	Mag	RC	Mag	RC	Mag	RC	Mag	RC	Mag
1A	.60	.5	10	1		0.5	2	38	M	38	M	36	M	36	M	30	M
1B	.60	.5	10	1		3		37	M	41	M	40	M	28	M	26	M
1C	.60	.5	10	1		3	2	40	M	45	M	45	M	41	M	35	M
2A	.40	.5	10	6		0.5	2	35	M	36	M	37	M	39	M	34	M
2B	.40	.5	10	6		3		21	SM	38	M	40	M	40	M	39	M
2C	.40	.5	10	6		3	2	35	M	44	M	45	M	45	M	46	M
2D	.40	.5	10	6		3	4	36	M	46	M	48	M	46	M	46	M
3A	.60	.5	13	1		0.5	2	29	M	36	M	35	M	30	M	26	M
3B	.60	.5	13	1		3		28	M	36	M	35	M	28	M	23	M
3C	.60	.5	13	1		3	2	30	M	40	M	40	M	42	M	31	M
4A	.40	.5	13	6		0.5	2	28	SM	27	SM	30	M	34	M	30	M
4B	.40	.5	13	6		3		22	NM	26	SM	31	SM	38	M	36	M
4C	.40	.5	13	6		3	1	27	SM	39	M	41	M	41	M	40	M
4D	.40	.5	13	6		3	2	27	SM	41	M	45	M	45	M	45	M
5A	.40	.5	15	6		0.5	2	23	NM	26	SM	30	M	32	M	28	M
5B	.40	.5	15	6		3		21	NM	23	SM	27	SM	39	M	39	M
5C	.40	.5	15	6		3	2	25	NM	36	SM	41	M	46	M	46	M
5D	.40	1.5	15	6	2.5	4	2	26	NM	33	SM	40	M	46	M	45	M
6A	.40	.5	18	4		0.5	2	26	M	38	M	36	M	34	M	30	M
6B	.40	.5	18	4		3		27	SM	40	M	38	M	36	M	33	M
6C	.40	.5	18	4		3	2	32	M	44	M	44	M	43	M	40	M
7A	.40	.5	18	6		0.5	2	22	SM	28	M	26	M	26	M	25	M
7B	.40	.5	18	6		3		22	NM	21	NM	27	SM	34	M	33	M
7C	.40	.5	18	6		3	2	21	M	33	M	40	M	40	M	40	M
7D	.50	.5	18	6		3	4	28	M	42	M	46	M	46	M	44	M
8A	.60	1.0	20	2		0.5	2	36	SM	38	M	35	M	33	M	31	M
8B	.60	1.0	20	2		3		44	NM	38	M	34	M	32	M	30	M
8C	.60	1.0	20	2		3	2	38	SM	40	M	42	M	39	M	37	M
9A	.40	.5	21	7		0.5	3	23	SM	24	SM	26	M	29	M	26	M
9B	.40	.5	21	7		2		23	NM	23	SM	21	SM	30	M	30	M
9C	.40	.5	21	7		2	3	25	M	35	M	40	M	39	M	39	M
10A	.50	.5	25	5		.70	2	23	M	23	M	27	M	26	M	25	M
10B	.40	.5	25	5		2		27	M	35	M	35	M	35	M	31	M
10C	.40	.5	25	5		2	2	38	M	45	M	46	M	46	M	42	M
10D	.40	2.5	25	5		3	2	27	M	33	M	40	M	50	SM	47	SM
11A	.40	2.5	26	7.5	1.25	0.5	2	22	M	26	M	33	SM	34	NM	31	NM
11B	.40	2.5	26	8	1.25	3		24	NM	26	NM	26	NM	30	NM	26	NM
11C	.40	2.5	26	7.5	1.25	2	2	27	M	38	SM	42	NM	41	NM	39	NM
12A	.60	3	30	8		0.7	2	24	SM	26	M	34	SM	36	NM	33	NM
12B	.60	3	30	8		3		22	SM	22	NM	37	NM	36	NM	37	NM
12C	.60	3	30	8		3	2	27	M	48	NM	50	NM	51	NM	50	NM

By way of illustration of the scope of my invention, other analyses having the desirable hard-

ening characteristics are given in the following table:

C	Mn	Cr	Ni	Cu	Si	Cb	2100° F. water quench		1250° F. 1 hour air cooled		1400° F. 1 hour air cooled		1400° F. 15 hours air cooled		1600° F. 1 hour air cooled	
							RC	Mag	RC	Mag	RC	Mag	RC	Mag	RC	Mag
.60	.5	10	1	----------	3	1	40	M	42	M	42	M	38	M	33	M
.40	.5	10	4	----------	3	2	33	SM	45	M	45	M	45	M	45	M
.40	.5	10	8	----------	3	2	30	SM	38	SM	40	M	46	M	50	M
.40	.5	10	10	----------	4	2	27	NM	29	NM	35	SM	46	M	46	M
.40	1.5	13	4	----------	3	2	30	M	42	M	43	M	43	M	42	M
.40	.5	13	4	----------	3	4	32	M	45	M	46	M	46	M	42	M
.40	.5	13	8	----------	3	2	25	SM	42	M	46	M	46	M	46	M
.40	.5	13	10	----------	4	2	26	SM	39	M	47	M	47	M	47	M
.60	.5	15	1	----------	3	2	28	M	38	M	38	M	37	M	35	M
.40	.5	15	4	----------	3	2	30	M	43	M	43	M	43	M	40	M
.40	1.5	15	4	2.5	4	2	28	NM	34	SM	40	M	45	M	44	M
.60	.5	15	6	----------	3	1	30	SM	33	SM	43	M	44	M	44	M
.60	.5	18	1	----------	3	2	30	M	35	M	38	M	38	M	37	M
.40	.5	18	8	----------	4	2	30	NM	37	SM	40	M	44	M	44	M
.60	1.0	20	2	----------	3	2	38	SM	40	M	42	M	39	M	37	M
.50	.5	20	5	----------	2.5	1	28	M	36	M	40	M	40	M	39	M
.50	.5	20	5	----------	2.5	3	30	M	40	M	42	M	43	M	42	M
.10	3	24	8	1.5	2	2	25	M	36	M	42	NM	42	NM	37	NM
.40	3	24	8	1.5	2	2	32	M	32	M	36	SM	37	NM	37	NM
.40	3	24	8	----------	2.5	3	29	SM	32	SM	38	SM	40	NM	38	NM
.10	2.5	24	7.5	----------	2	2	19	M	37	M	44	SM	46	NM	43	NM
.40	.5	25	7	Ta 1.65	2	1.35	29	M	35	M	40	M	47	NM	40	SM
.10	.5	25	7	----------	2	3	39	M	48	M	50	M	44	M	44	M
.40	2	27	5	----------	2	2	28	M	37	M	44	M	46	NM	45	SM
.40	2	27	2	----------	3	3	24	M	30	M	37	M	39	M	37	M
.40	3	27	9	----------	3	5	29	M	53	NM	60	NM	60	NM	60	NM
.40	2	30	5	----------	2	2	24	M	36	M	47	NM	47	NM	46	SM
.40	3	30	8	----------	3	2	27	M	48	NM	50	NM	51	NM	50	NM
.50	2	35	5	----------	3	2	26	M	56	NM	58	NM	58	NM	56	NM
.50	2	35	16	----------	3	3	28	SM	47	NM	48	NM	54	NM	54	NM

The analyses in the tables are what are known as heat analyses. Many of these heat analyses were subjected to chemical analysis and there does not seem to be much difference between the actual chemical analysis and the heat analysis, and for clarity the heat analysis has been given. On chemical analysis, the alloys will be about .5% or less lower in chromium than given in the tables. The silicon content will be within about .5% of the analysis given, generally closer than this. The columbium content will be substantially that of the heat analysis, and this is true of the nickel. Where copper is employed, practically none is lost in melting. The carbon content will be within about .05% of the figure given for the heat analysis for medium carbons, and considerably less for low carbons. In the tables, in addition to the analyses of the steel, I have shown the Rockwell C hardness and the state of magnetism of each alloy when given a water quench from 2100° F., when air cooled after being held at a temperature of 1250° F. for one hour, air cooled after being held at 1400° F. for one hour, air cooled after being held at 1400° F. for 15 hours and air cooled after being held at 1600° F. for one hour. In the columns designating the magnetism, the letter M indicates that the alloy is magnetic, NM indicates it is non-magnetic and SM indicates it is slightly magnetic.

The tables have been restricted to preferred ranges, and with the chromium content over 23%, nickel lower than 5% gives excellent hardness, but the alloys are less tough with the lower nickel, and also lacking in strength at red heat. Alloys containing 18% chromium and less have excellent strength at red heat, even though the nickel may be low. They, too, however, increase in strength at red heat as the nickel is increased.

The columbium analysis given in the tables is given in many instances in the order of about 2%. It represents a desirable percentage. More columbium can be employed. Over 3%, although it does increase the hardness, ordinarily is not justified because of the increased cost. Under these circumstances the preferable range of columbium is between 1% and 3%, and for many instances, and particularly for valves for internal combustion engines as little as .5% of columbium is an advantage.

In the tabulation it will be noted that all of the examples embodying my invention show a Rockwell C hardness of at least about 30 and most of the examples show a Rockwell C hardness of 35 or more, after the steel has been heated to temperatures in the order of about 1400° F. The steels having substantially lower hardness are those which were made for the purpose of checking to show that either the presence of the columbium with low silicon or the presence of high silicon alone would not give a hardness which is obtainable by the combination of the two.

I am not restricting my invention to the examples given. They serve thoroughly to describe and depict the hardness properties of the alloys within their very useful range. For special purposes the analysis can be modified within the range of my invention and the properties of the alloys retained with slight modifications that can be readily seen from the tables herein included.

The iron component of my various alloys naturally will contain the usual impurities common to commercial alloy steels, and these, together with the permissible percentages of high melting point elements referred to above are to be considered as included when in the claims I refer to the fact that in addition to the named alloying ingredients the balance is principally iron.

The alloys of my invention not only are easily hardenable, but they can also be readily softened by cooling down or quenching from high temperatures, and in the softened state, they are easily machineable and have excellent cold malleability. These characteristics, plus the fact that they forge well, means that various articles can be made from these steels at a reasonable expense. Also my alloys can advantageously be used for making castings, for when melted with reasonable care and skill, they cast solid and are quite free from occluded gases.

The outstanding value of the steels of my invention relate to their use under conditions where they may be maintained at a red heat, that is, at temperatures ranging from about 1100° F. up to as much as 1600° or 1800° F. In this range the alloys maintain the hardness which has already been specified and they have more than usual strength and very excellent creep resistance. Also they have excellent resistance to scaling at these high temperatures.

The effect of silicon on scale resistance is well known, but apparently this effect is increased by the combination of columbium with silicon. Due to the fact that hardness can be obtained independently of the carbon content, my alloys, particularly those high in chromium, can be produced which are amply hard for most uses and yet where inter-granular corrosion would normally be expected, they are particularly resistant to this form of attack.

These characteristics indicate that the alloys of this application are particularly valuable, as stated, for various articles to be used and maintained at temperatures that may come into a red heat range, such for example, as valves or valve elements for internal combustion engines, turbine blades to be used with superheated steam, chemical equipment and the like. For example, in the case of turbine blades, usually austenitic steels are not satisfactory for they are too soft and subject to twisting and eroding at the edges. A steel such as those of my invention having the corrosion resistance of the usual austenitic stainless steels but having a Rockwell "C" hardness of 35, or better, at working temperatures, and having high strength at those temperatures, is a distinct advance. In the same way, the steels of my alloys can advantageously be used for piston rods where strength is of great importance.

What I claim is:

1. An article at least a portion of which is made of a ferrous alloy steel characterized by the fact that it has been hardened to a hardness of at least about 30 on the Rockwell "C" scale at a temperature within the approximate range of 1200° F. and 1800° F. and further characterized by its strength and resistance to corrosion at red heat, said alloy steel containing a plurality of alloying ingredients of which the following, in the proportions stated, are the only elements necessary to attain said characteristics: chromium between about 10% and 35%, nickel between about 1% and 16%, silicon between about 1% and 5%, columbium between about .5 and 5%, and manganese ranging up to 4%.

2. A valve or valve element for internal combustion engines as specified in claim 1.

3. An article as specified in claim 1, in which the chromium range is between 10% and 20%, the nickel range between about 1% and 8%, the silicon range between about 2% and 4% and which also contains between about .3% and 3% of manganese.

4. An article as specified in claim 1, in which the chromium range is between 13% and 18%, the nickel range between 4% and 6%, the silicon range between 2% and 4%, the columbium range between 1% and 3% and which also contains between .50% and 1.5 of manganese.

5. An article as specified in claim 1, in which the chromium range is between 20% and 23%, the nickel is between about 2% and 10%, the silicon is between about 1.5% and 4%, and which also contains manganese between .3 and 4%.

6. An article as specified in claim 1, in which the chromium range is between 20% and 23%, the nickel range between 2% and 10%, the silicon range between 1.5 and 3.5%, the columbium range between 1% and 3% and which also contains between 1% and 3% of manganese.

7. An article as specified in claim 1, in which the chromium range is between 23% and 35%, the nickel range is between about 2% and 16%, the silicon range is between about 1.5% and 4% and which also contains manganese between about .3% and 4%.

8. An article as specified in claim 1, in which the chromium range is between 23% and 30%, the nickel range between 2% and 10%, the silicon range between 1.5% and 3.5%, the columbium range between 1% and 3%, and which also contains between 1% and 3% of manganese.

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