Vacuum heat treatment

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Catalogue

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Preface

Vacuum heat treatment is a heat treatment process in which the workpiece is heated to the desired temperature in a 101Pa vacuum medium and then cooled at different cooling rates in different media.

Vacuum heat treatment is called clean heat treatment with high efficiency ,energy saving and no pollution.Vacuum heat treatment parts have a series of advantages, such as no oxidation, no decarbonization, degassing, degreasing, good surface quality, small deformation,high comprehensive mechanical properties, good reliability (good repeatability, stable life) and so on. Therefore, the vacuum heat treatment has received extensive attention and widespread application at home and abroad. Taking the popularity of vacuum heat treatment as an important symbol to measure the level of heat treatment technology in a country. Vacuum heat treatment technology is a hot spot of heat treatment technology development in the past 40 years, and it is also an important field of advanced manufacturing technology.

I.The principle of vacuum heat treatment and the characteristics of vacuum heat treatment and heating

1.Technical principle

(1)The phase transition characteristics of gold net under vacuum.

The thermodynamics and kinetics of solid-state phase change do not change much in vacuum with atmospheric pressure difference of 0.1MPa. When formulating the vacuum heat treatment process rules,can be completely based on the principle of solid phase transformation under atmospheric pressure, and the data of various types of tissue transformation under atmospheric pressure.

(2) The effect of vacuum degassing is to improve physical and mechanical properties of metallic materials.

(3) The effect of vacuum degassing.

(4) Evaporation of metals:heating in vacuum, the elements on the surface of workpiece will produce the evaporation phenomenon

Table1 vapor pressure of various metals

Metal	The equilibrium temperature which reach the vapor pressure below (°C)					Melting point(°C)
	10^{-2}Pa	10^{-1}Pa	1Pa	10Pa	133Pa	
Cu	1035	1141	1273	1422	1628	1038
Ag	848	936	1047	1184	1353	961
Be	1029	1130	1246	1395	1582	1284
Mg	301	331	343	515	605	651
Ca	463	528	605	700	817	851
Ba	406	546	629	730	858	717
Zn	248	292	323	405	-	419
Cd	180	220	264	321	-	321
Hg	-5.5	13	48	82	126	-38.9
Ae	808	889	996	1123	1179	660
Li	377	439	514	607	725	179
Na	195	238	291	356	437	98
K	123	161	207	265	338	64
In	746	840	952	1088	1260	157
C	2288	2471	2681	2926	3214	-
Si	1116	1223	1343	1485	1670	1410
Ti	1249	1384	1546	1742	-	1721
Zr	1660	1861	2001	2212	2549	1830
Sn	922	1042	1189	1373	1609	232
Pb	548	625	718	832	975	328
V	1586	1726	1888	2079	2207	1697
Nb	2355	2539	-	-	-	2415

Ta	2599	2820	-	-	-	2996
Bi	536	609	693	802	934	271
Cr	992	1090	1205	1342	1504	1890
Mo	2095	2290	2533	-	-	2625
Mn	791	873	980	1103	1251	1244
Fe	1195	1330	1447	1602	1783	1535
W	2767	3016	3309	-	-	3410
Ni	1257	1371	1510	1679	1884	1455
Pt	1744	1904	2090	2313	2582	1774
Au	1190	1316	1465	1646	1867	1063

（5） The effect of surface purification, to achieve less oxidation and less no decarbonization heating.

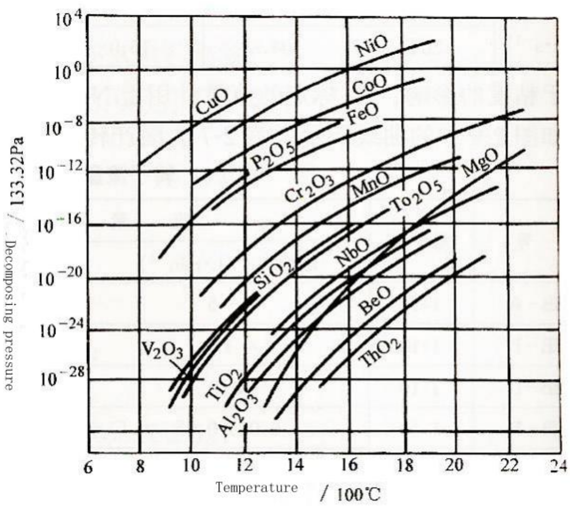

Figure 1 decomposition pressure of various metal oxides

The oxidation reaction of metals is reversible:Mo≒2M+2O　2O→O2↑

It depends on the partial pressure of oxygen and the partial pressure of metal oxid es.

When the partial pressure of oxygen is greater than the partial pressure of metal o xide, the reaction proceeds to the left and the metal surface produces oxidation. On t he contrary, if the decomposition pressure of oxide is greater than the partial pressure of oxygen, the reaction proceeds to the right, and the result is oxide decomposition.

The theory of protoxide and the existence of carbon element in vacuum furnace make the partial pressure of oxygen in furnace lower than that of metal oxide, so that metal will not be oxidized.

Table two relationship between vacuum and relative impurities and relative dew point

Vacuum degree	Pa	1.33×10^{4}	1.33×10^{3}	1.33×10^{2}	1.33×10	1.33	1.33×10^{-1}	1.33×10^{-2}	1.33×10^{-3}
	torr	100	10	1	10^{-1}	10^{-2}	10^{-3}	10^{-4}	10^{-5}
Relative impurity conten	%	13.2	1.32	0.132	1.32×10^{-2}	1.32×10^{-3}	1.32×10^{-4}	1.32×10^{-5}	1.32×10^{-6}
	PPM(part per millio			1320	132	13.2	1.32	0.132	0.0132

t	n)								
Relative dew point（°C）			+11	-18	-40	-59	-74	-88	-101

(5) The degree of vacuum required for non oxidizing heating of a metal.

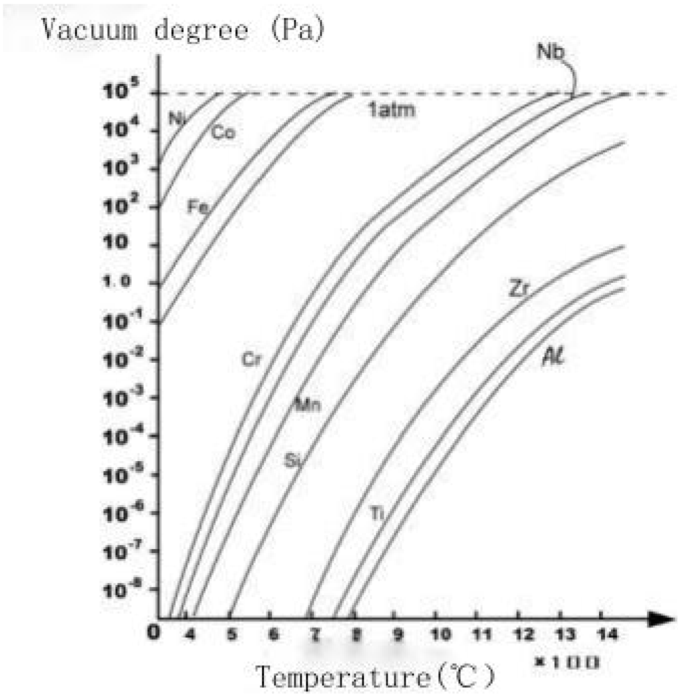

Figure 2 the relationship curve between heating temperature and vacuum degree of different metals.

2.Heating characteristics of vacuum heat treatment

Two remarkable features: one is when the no-load is heated, the heating speed is fast, and the two is the slow heating speed of the workpiece .

II.Determination of technological parameters for vacuum heat treatment.

1.vacuum degree:

Table 3 vacuum degree of various materials in vacuum heat treatment.

materials	vacuum degree of vacuum heat treatment (Pa)
Alloy tool steel, structural steel and bearing steel (quenching temperature below 900 ℃).	$1 \sim 10^{-1}$
Containing Cr, Mn, S and other alloy steels (heating above 1000℃).	10Pa（backfill high purity nitrogen）
Stainless steel (precipitation hardening type gold alloy), Fe、Ni based alloy, cobalt based alloy	$10^{-1} \sim 10^{-2}$
titanium alloy	10^{-2}
High speed steel	1000℃above fill in 666~13.3Pa N_2
Cu and its alloys	133~13.3Pa
Tempering of high alloy steel	$1.3 \sim 10^{-2}$

We should pay attention to several points when considering working vacuum.

(1) before 90℃, high vacuum above 0.1Pa should be pumped for degassing.

(2) Heating in 10-1Pa， which is equivalent to the purity of inert gas above 1PPM.most of black metal will not oxidize.

(3) When filled with inert gas,such as 133Pa, (50%N2+50%H2) nitrogen and ammonia mixture gas, the effect is better than 10-3Pa vacuum. The oxygen partia l pressure 66.5Pa is safe at this time.

(4) the degree of vacuum corresponds to the brightness of steel surface.

(5) Generally speaking, in the vacuum range of 10-13 Pa, the temperature

difference of vacuum degree is±5℃. If the air pressure rises, the temperature uniformity decreases, so the inflation pressure should be as low as possible.

2.heating and preheating temperature:

Table 4 reference list for preheating temperature

Quenching heating temperature 温度（℃）	Preheating temperature（1）（℃）	Preheating temperature（2）（℃）	Preheating temperature（3）（℃）
800~900	550-600		
1000-1100	550-600	800-850	
Above 1200	550-600	800-850	1000-1050

3.Vacuum quenching heating time

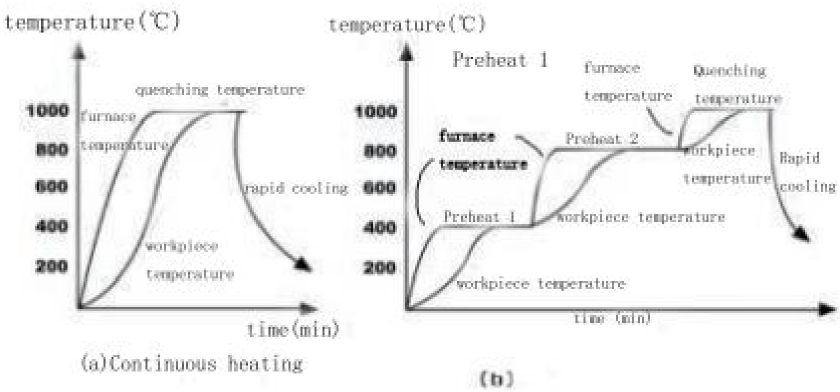

Figure 3 characteristic curve of vacuum heating

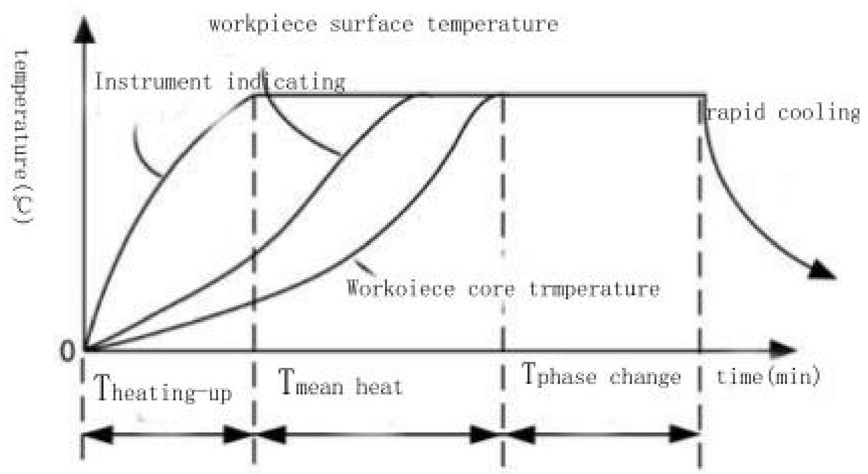

Figure 4 furnace temperature and the surface and central temperature of the heated workpiece

$T_{total} = t_{mean\ heat} + t_{phase\ change}$ $t_{mean\ heat} = a` \times h$

$t_{mean\ heat}$ is time of mean heat， $t_{phase\ change}$ is time of phase change， a` is diathermy coefficient（minute/mm）， h is effective thickness（mm）

Table 5 a`diathermy coefficient of certainty

Heating temperature（℃）	600	800	1000	1100~1200
a`（minute/mm）	1.6~2.2	0.8~1.0	0.3~0.5	0.2~0.4
Preheating condition		600℃preheating	600、800℃preheating	600、800、1000℃preheating

Notice:without preheating and direct heating ,a` should increase 10~20%

Table 6 $t_{phase\ change}$ time determination

Steel products	Carbon tool steel	Low alloy steel	High alloy steel
$t_{phase\ change}$（minute）	5~10	10~20	20~40

III.The cooling method of vacuum heat treatment

1.Gas quenching

(1) Properties of various cooling gases

Table 7 Properties of various cooling gases

Gas	Density (Kg/m³)	Prandt number	Viscosity coefficient (Kg.s/m³)	Thermal conductivity (kcal/m.h. ℃)	Thermal conductivity ratio
N_2	0.887	0.70	2.5×10^{-6}	0.0269	1
Ar	1.305	0.69	2.764	0.0177	0.728
He	0.172	0.72	2.31	0.143	1.366
H_2	0.0636	0.69	1.048	0.189	1.468

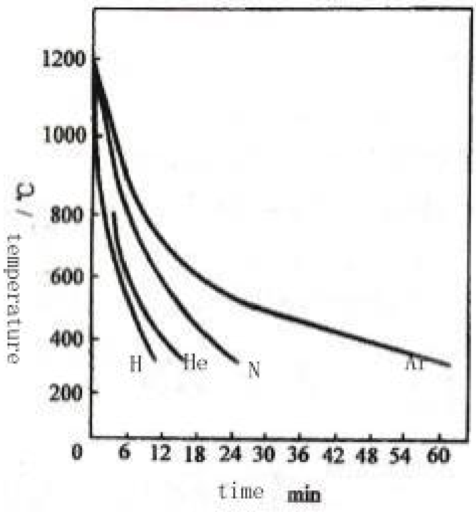

Figure 5 relative cooling performance of hydrogen, helium, nitrogen and argon

In order to ensure that the workpiece surface is non oxidized and has high brightn ess, it has certain requirements for the purity of cooling gas N2.

Table 8 nitrogen purity standard

Processing material	nitrogen purity （%）
Bearing steel and high speed steel	99.995~99.998
High temperature heat resistant alloy	99.999
High temperature active metal	99.9999

Semiconducting material	99.99999

Table 9 occupation standard of argon、hydrogen and nitrogen for heat treatment

Name		Target requirement，%（V/V）					
		Argon content	Nitrogen content	Hydrogen content	Oxygen content	Total carbon content（calculated in terms of methane）	Water content
High purity argon		≥99.999	≤0.0005	≤0.0001	≤0.0002	≤0.0002	≤0.004
argon		≥99.99	≤0.007	≤0.0005	≤0.001	≤0.001	≤0.002
High purity nitrogen		-	≥99.999	≤0.0001	≤0.0003	≤0.0003	≤0.0005
Pure nitrogen		-	≥99.996	≤0.0005	≤0.001	CO≤0.0005 CO_2≤0.0005 CH_4≤0.0005	≤0.0005
Gaseous nitrogen for industrial use	Class I	-	99.5	-	≤0.5	-	Dew point≤-43℃
	Class IIlevel I	-	99.5	-	≤0.5	-	freewater≤100ml/bottle
	Class IIlevel II	-	98.5	-	≤1.5	-	freewater≤100ml/bottle
hydrogen		-	≤0.006	≥99.99	≤0.0005	CO≤0.0005 CO_2≤0.0005	≤0.003

					CH_4≤0.001	

Notice :1.water partial pressure 15 ℃,determined under the condition of greater than 118 MPa.

2.High purity nitrogen、pure nitrogen are not suitable for vacuum heat treatment refilling and cooling gas of precipitation hardening stainless steel, maraging steel, high temperature alloy, titanium alloy and so on.

3.Hydrogen is not suitable for heat treatment protection of high strength steel, titanium alloy and brass.

4.Liquid nitrogen does not specify the amount of water.

(2) Ways to improve gas cooling capacity

Newton formula：$Q=k(t_w-t_f)\cdot F$（kcal/h）

Q is heat transfer； t_w is workpiece temperature； t_f is gas temperature；

F is the surface area of the workpiece；

k is the convective heat transfer coefficient。

$$K=(\lambda/d)\cdot C(wdp/\eta)^{m}$$

d is diameter of workpiece， **C** is constant that varies with the range of Reynolds coefficients， **m** is power exponent，generally is 0.62~0.805

w is velocity of flow，**p** is a function of density (also known as pressure)，

λ is thermal conductivity of the gas，**η** is viscous coefficient.

It can be seen from the formula that increasing the density (pressure) and velocity of cooling gas can increase the efficiency of heat transfer in proportion.

①Increase the cooling gas pressure

②Increase the flow rate of the gas

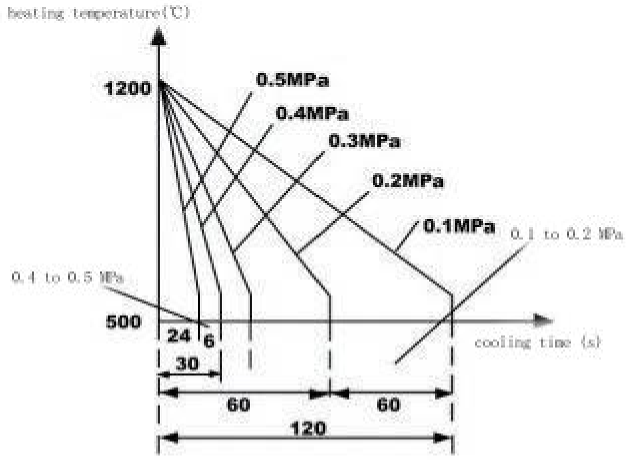

Figure 6 relationship curve between gas pressure and quenching rate

Table 10 comparison of coefficient of heat conduction between various quenching media

Medium and quenching parameters	Heat conductivity（$w/m^2.k$）
Salt bath 550℃	350~450
Liquid bed	400~500
Oil 20~80℃ doesn't flow	1000~1500
Oil 20~80℃ stirring cycle	1800~2200
Water 15~25℃	3000~3500
Air 、no brute force circle	50~80
1000mb（1×10^5Pa）N_2 cycle	100~150
6×10^5Pa N_2 fast cycling	300~400
10×10^5Pa N_2 fast cycling	400~500
6×10^5Pa He fast cycling	400~500
10×10^5Pa He fast cycling	550~650
20×10^5Pa He fast cycling	900~1000
6×10^5Pa H_2 fast cycling	450~600
10×10^5Pa H_2fast cycling	~750
20×10^5Pa H_2 fast cycling	~1300
40×10^5Pa H_2 fast cycling	~2200

2.Vacuum oil quenching

(1) Condition for vacuum quenching

(2) Main technical indexes of vacuum quenching oil

Table 11 (a) quality index of vacuum quenching oil made in China

Vacuum quenching code	ZZ-1	ZZ-2
Viscosity （cst）50℃	20~25	50~55
Flash point（℃）not less than	170	210
Freezing point（℃）not higher than	-10	-10
Water （%）	Nothing	Nothing
Residual carbon（%）not greater than	0.08	0.1
Acid value（mgkoH/g）	0.5	0.7
The saturated vapor pressure 20℃（133Pa）	5×10^{-5}	5×10^{-5}
Thermal oxidation stability	Qualified	Qualified
Cooling performance Characteristic temperature （℃）	600~620	580~600
characteristic time（s）	3.0~3.5	3.0~4.0
The time needed to cool at 800℃ to 400℃（s）	5~5.5	6~7.5

Table 11 (b) Quality index of vacuum quenching oil of Shanghai Huifeng Petrochemical Co., Ltd.

<table>
<tr><th>Item /Model</th><th>CZ1 vacuum quenching oil</th><th>CZ2 vacuum quenching oil</th><th>Test method</th></tr>
<tr><td>Kinematic viscosity（40℃）， mm^2/s</td><td>32～42</td><td>80～90</td><td>GB/T265</td></tr>
<tr><td>Flash point（开口）， ℃</td><td>180</td><td>220</td><td>GB/T3536</td></tr>
<tr><td>Pour point ， ℃</td><td>-10</td><td>-10</td><td>GB/T3535</td></tr>
<tr><td>Cooling performance
Characteristic temperature（℃）
The time needed to cool at 800℃ to 400℃（s）</td><td>600
5.5</td><td>585
7.5</td><td>SH/T0220</td></tr>
<tr><td colspan="4">Note： The above data are the test results of representative samples, and the product performance shall be subject to the actual test</td></tr>
<tr><td colspan="4">Performance ： 1.Low saturated vapor pressure and small evaporation,so that the dissolved gas quickly out；
2.Strong anti-vaporization ability and faster cooling speed, no pollution to the vacuum furnace and vacuum operation effect
3.Stable cooling performance, in vacuum conditions, can ensure that t he quenched workpiece hardening effect is good.
4.Good brightness , clean and bright after quenching, no discoloration, no oxidation, no pollution
5.Excellent volatile stability and oxidation stability, long service life.</td></tr>
<tr><td colspan="4">Application: 1. It is suitable for bearing steel, tool die, cutting tools, large and medium sized aviation structural steel and other special steels.
2.HFY-CZ1 vacuum quenching oil is used to quench medium-sized materials in vacuum, HCZ2 vacuum quenching oil is used to quench materials with good permeability in vacuum.</td></tr>
</table>

Table 12 quality index of vacuum quenching oil of C.I.Hayes , USA

Vacuum quenching oil code	H_1	H_2
Specific gravity（Ib/gal）	7.36	7.2
Viscosity index	76	95
viscosity（100°F）sus	92~95	110~121
Lgnition point（°C）	170	190
Hot-wire test	34.0	31.0
Vapour pressure 40°C（133Pa）	0.002	0.0001
90°C（133Pa）	0.100	0.0103
150°C（133Pa）	2.00	0.45
GM quenching test（s）	11	17
The highest temperature（°C）	60	80

Several questions that should be noticed when vacuum oil quenching is used :

1. Vacuum oil quenching pressure is filled with pure M240kPa-67kPa.

2.The quantity of quenching oil: workpiece : oil weight 1：10~15,The oil pool is 15~20% larger than the sum of oil and workpiece volume

3.No water is allowed in the oil. When the workpiece reaches 0.03%, the workpiece becomes darker and the cooling rate changes obviously at 0.3%.

4.The concoct of vacuum quenching oil

5.The gas should be fully degassed before the workpiece entering the oil.

6.Oil should be used under the temperature of 40~80°C

7.Oil should be stirred. The static oil cooling strength is 0.25-0.30; the intensity of the heated mixing oil is 0.8-1.1.

8.The phenomenon of high temperature instant acieration in vacuum oil quenching.

3.stage cooling is used to reduce workpiece deformation

1.Oil is cooled to more than M_3 point → air patenting

2.Delay oil quenching , pre cooling 30~70 seconds →（1090°C）add oil

3.Air patenting to 550°C→ quenching in the oil

4.Gas stage cooling , gas cooling to martensite point above→stop the fan→turn on the fan for cooling fast after the surface temperature is uniform.

5.The workpiece is isothermal quenching in nitrate bath.

4.Vacuum water quenching

5.Vacuum nitrate quenching.

6.Furnace cooling or controlled cooling.