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Martensitic and magnetic transformation of $Co_{41}Ni_{32}Al_{24}Sb_3$ and $Co_{41}Ni_{32}Al_{27}$ alloys

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Abstract: The martensitic transformation and magnetic property of $Co_{41}Ni_{32}Al_{27}$ and $Co_{41}Ni_{32}Al_{24}Sb_3$ alloys were investigated by optical microscopy(OM), scanning electric microscopy(SEM), energy dispersive X-ray spectroscopy(EDS), X-ray diffractometry (XRD), differential scanning calorimeter analysis(DSC) and vibration sample magnetometer(VSM) methods. The results show that martensitic crystal structure of $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy is still $L1_0$ type. Both martensitic transformation temperature T_m and Curie point T_c are in linear relation to quenching temperature. T_m increases by 9 K and T_c increases by 7.5 K for every 10 K increasing in quenched temperature. Quenched from same temperature, T_m of $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy is higher than that of $Co_{41}Ni_{32}Al_{27}$ alloy by 76 K, meanwhile T_c is higher by 18 K. The melting point of Co-Ni-Al alloy is decreased by the Sb addition, eutectic structure appears in $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy annealed at 1 573 K, which indicates that the alloy is partially melted, whereas $Co_{41}Ni_{32}Al_{27}$ alloy can be annealed at 1 623 K without melted. The martensitic transformation temperature range of $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy is 22−29 K, only half that of $Co_{41}Ni_{32}Al_{27}$ alloy. This is a very important result to benefit the achievement of large magnetic field induced strain on Co-Ni-Al based alloy. The results of T_m and T_c were explained by total average s+d electron concentration and magnetic valence number Z_m respectively.

Key words: ferromagnetic shape memory alloy; Co-Ni-Al-Sb alloy; martensitic transformation; Curie point

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1 Introduction

β-base Co-Ni-Al ferromagnetism shape memory alloys are widely noticed because of their good hot/cold workability[1,2]. β-base Co-Ni-Al alloys have the B2→$L1_0$ phase transformation[3,4], the T_c of the alloys can be increased by the increasing Co content. When the T_c is higher than the martensitic phase transformation temperature, the martensitic phase transformation can be induced by the magnetic field and the ferromagnetism shape memory alloys are produced. The Co-Ni-Al alloys are classical excellent shape memory alloys[5,6]. But the shape change of these alloys is only 0.06%[7], while that of Ni_2MnGa alloys is as large as 6% magneto strain[8]. With respect to the martensitic phase transformation, if there is no external stress, in the Co-Ni-Al alloys only the $L1_0$ type martensitic is formed while in the Ni_2MnGa alloys the 10M and 14M types appear either. The range of the martensitic phase transformation of the Co-Ni-Al alloys is as large as 100 K while that of Ni_2MnGa is a narrow temperature range[9]. In order to get large magneto strain on the base of Co-Ni-Al alloys, the fourth content is added to change the phase transformation and the magnetic properties[10]. The substitution of the element Ga with element Sb in the Ni-Mn-Ga alloys results in formation of the Heusler Ni-Mn-Sb alloys in which the multiple phase transformation is similar to that of the Ni-Mn-Ga remains [11]. In the paper the element Sb is added to the β-base Co-Ni-Al alloys to study the effect of the substitution of part Al with Sb on the martensitic phase transformation and the T_c of the Co-Ni-Al alloys.

2 Experimental

$Co_{41}Ni_{32}Al_{27}$ and $Co_{41}Ni_{32}Al_{24}Sb_3$ alloys were prepared by ingot metallurgy with pure metal. Melting was carried out with an arc-melting furnace by protected argon. The mass of each button-like ingot was 30 g. In order to ensure composition is homogeneous, each sample was inverted and remelted five times. Initial

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samples were sealed in quartz tube by protective argon gas, $Co_{41}Ni_{32}Al_{27}$ alloy was annealed at 1 623 K for 24 h, and $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy was annealed at 1 523 K for 24 h. They were cooled in air. Then, they were cut small, and were sealed in quartz tube again. The samples were quenched in water after isothermal treating at 1 423 K for 24 h, 1 473 K for 24 h, 1 523 K for 12 h, 1 573 K for 4 h, 1 623 K for 2 h, respectively. Composition of each phase was close to balance state on thermodynamic equilibrium, treat-time of samples were protracted at lower temperature. The $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy was not analysed, because it was melted seriously at 1 623 K for 2 h.

Optical microscope(OM) and scanning electron microscope(SEM) images were taken using Nikon Epiphoto 200 and Philips XL 30S FEG equipped, and the phase compositions of alloys were measured using CDULEAP energy dispersive X-ray spectroscopy (EDS) apparatus on SEM. The X-ray diffraction spectra were measured on Philips X'pert MPD X-ray diffractometer with monochromated Cu K_α. The temperature of martensitic transformation was measured using a Seiko Exstar DSC6200 apparatus at a heating rate of 10 K/min. The original and final temperature (M_s and M_f) of martensitic transformation were obtained among temperature reduced by measuring continuously. The original and final temperature (A_s and A_f) of martensitic transformation inverse were obtained among temperature promoted by measuring continuously. Curie point(T_c) of ferreous magnetic transformation were taken using Toei VSM-5 equipped by measuring curve of magnetic intensity(M) with temperature(T), Specimens of being used liquefaction nitrogen cooling were measured among 90−473 K with a heating rate of 2 K/min when outer-magnetic intensity is 4×10^4 A·m^{-1}. In order to ensure that the results were comparable, the specimens were used as that of DSC.

3 Results and analysis

Under the thermodynamic equilibrium, with the enhancement of the heat treatment temperature, in the $\beta+\gamma$ Ni-Al[12,13] and the Co-Ni-Al[2,4] alloys, the volume fraction of β increases and the volume fraction of γ decreases. The metallurgical structure of the $Co_{41}Ni_{32}Al_{27}$ reflects this regular. The alloy is firstly heat treated at 1 623 K for 24 h and the β grains and the precipitated γ are coarse (Fig.1(c)). When the quenching temperature is decreased to 1 573 K the fine needle-like γ precipitates (Fig.1(b)), and the lower the quenching temperature, the finer and the more γ precipitates (Fig.1(a)). From Fig.1(d) and Fig.1(e), it can be seen that the volume fraction of the γ decreases with the increase of the quenching temperature, and the shape of the γ is obviously different and the new γ is round. It can be found from Fig.1 that the matrix of the $Co_{41}Ni_{32}Al_{27}$ is lath-shape martensite when quenched at 1 623 K, and the matrix of the $Co_{41}Ni_{32}Al_{24}Sb_3$ is already lath-shape $Co_{41}Ni_{32}Al_{24}Sb_3$ when quenched at 1 523 K.

From Fig.1(f), it can be seen that the matrix of the $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy is lath-shape or lamellar martensitic, but the matrix is spheroidized grains and white structure appears both at the grain boundary and in the interior of the grains. In Fig.2, the structures quenched at 1 523 K and 1 573 K are compared and the white structure quenched at 1 573 K is of the characteristic of eutectic which is composed of two phases: β and γ. This indicates that the eutectic temperature of this alloy is lower than 1 537 K and it melts partly at 1 573 K. Till 1 623 K, the $Co_{41}Ni_{32}Al_{27}$ does not melt, which indicates that the substitution of Al with Sb decreases the melting point of the Co-Ni-Al.

The decrease of the melting point benefits the production of the single crystal which is important to the study and application to FSMA because the maximum shape change in the magneto strain is gotten through driving the martensite variants to arrange in preferred orientation under external magnetic field. Due to low melting points, the single crystal of the Ni_2MnGa and Ni-Fe-Ga alloys can be produced highly efficiently by Bridgman single crystal furnace. While with relatively higher melting points, the $Co_{41}Ni_{32}Al_{27}$ does not melt at 1 653 K and the temperature of complete melting is much higher, so its single crystal should be produced using optical floating zone[7,14]. The latter method is capital-expensive, inefficient and needs persons to monitor, which bring difficulty to production. The addition of the Sb to Co-Ni-Al decreases the melting point and it is possible to produce single crystal using the Bridgman method.

Fig.3 shows the XRD pattern of the $Co_{41}Ni_{32}Al_{24}Sb_3$ alloys. This kind of alloys will result in more precipitants of γ and decrease the martensite phase transformation temperature[10] which takes difficulty to the determination of the crystal structure, so equenched plate $Co_{41}Ni_{32}Al_{24}Sb_3$ was used for XRD. From Fig.3, the martensitic of the $Co_{41}Ni_{32}Al_{24}Sb_3$ alloys quenched at 1 523 K is of the $L1_0$ structure, the γ is face-centered cubic which accords with that of the CoNiAl alloys[1,4,10]. There are a few XRD peaks of parent phase of the B2 type.

As shown in Fig.4, in the temperature range of the DSC conducted, thermal enthalpy peaks appear in the DSC curves of the seven samples except for the $Co_{41}Ni_{33}Al_{27}$ quenched at 1 423 K and 1 473 K. The temperatures of the M_s, M_f, A_s, A_f of each sample are calibrated through standard DSC and marked with

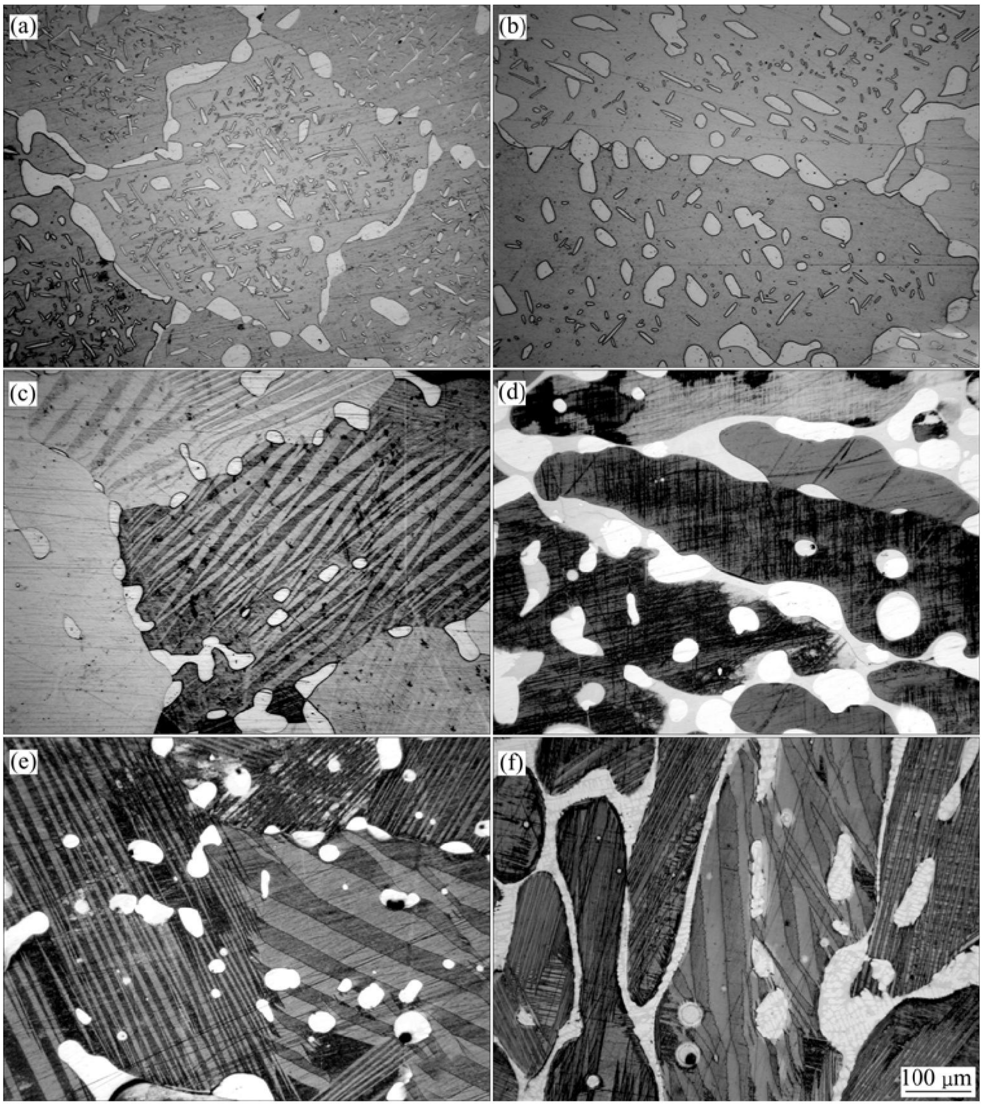

Fig.1 Metallographs of $Co_{41}Ni_{32}Al_{27}$ alloy annealed at 1 523 K (a), 1 573 K (b), 1 623 K (c) and $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy annealed at 1 473 K (d), 1 523 K (e) and 1 573 K (f)

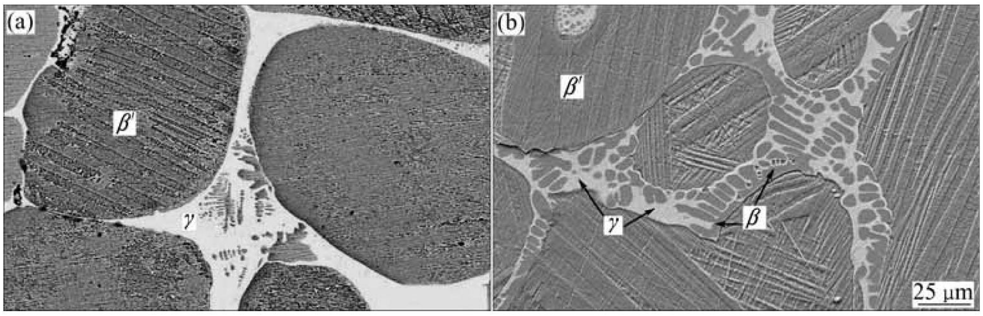

Fig.2 SEM pictures of $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy annealed at 1 523 K (a) and 1 573 K (b)

arrows in Fig.4. The specified martensite phase transformation temperatures T_m are listed in Table 1. The T_m of both alloys decreases with the lowering quenching temperature, and at the same quenching temperature the T_m of the $Co_{41}Ni_{32}Al_{24}Sb_3$ is higher than that of the $Co_{41}Ni_{32}Al_{27}$. The T_m of the $Co_{41}Ni_{32}Al_{24}Sb_3$ quenched at 1 523 K is higher than room temperature (293 K), while in the $Co_{41}Ni_{32}Al_{27}$ alloy system, only the T_m of that quenched at 1 623 K is higher than room temperature. All these accord well with the metallographic analysis.

The thermomagnet M—T curves are shown in Fig.5, T_c is the temperature corresponding to the minimum of the $\partial M/\partial T$—T curve which is the inflection temperature of the M rapid decreasing stage in the M—T curve. The

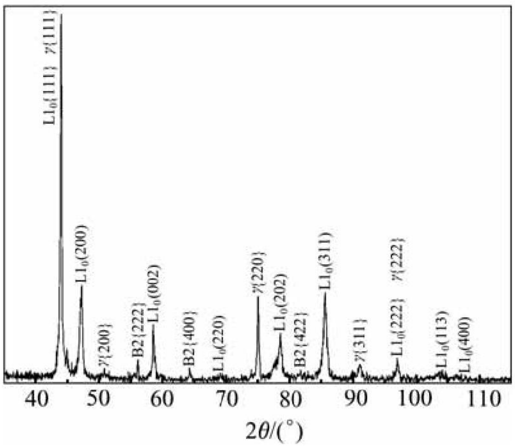

Fig.3 XRD pattern of $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy plate quenched after heat treatment at 1 523 K for 12 h

T_c of each sample is marked in Fig.5 and they are listed in Table 1 also. The M—T curves can serve to determine the inverse martensitic transformation and the M increases with T in some district which corresponds to the start temperature A_s and the final temperature A_f of the inverse martensitic transformation. The A_s and A_f of some alloys are also listed in Table 1 and they accords with that measured through DSC. The M—T curves of the $Co_{41}Ni_{33}Al_{27}$ quenched at 1 423 K and 1 473 K also have this kind of process, which indicates that martensitic transformation occurs and the corresponding A_s and A_f are listed in Table 1.

The temperature range of the martensitic transformation is represented as $\Delta T_m=((M_s-M_f)+(A_f-A_s))/2$ in Table 1. The results show that the ΔT_m of the $Co_{41}Ni_{32}Al_{27}$ is 40−55 K but that of the $Co_{41}Ni_{32}Al_{24}Sb_3$ is 22−29 K, which is only half that of the $Co_{41}Ni_{32}Al_{27}$. These results are important to the development of FSMA with high magneto strain. The important principle of FSMA is the shape change generated from martensitic transformation induced by external magnetic field which is to say martensitic transformation induced by stress. The martensitic transformation induced by stress occurs only in some temperature range which is some little higher than M_s, and when the force-applying temperature rises the force will increase until the martensitic transformation does not occur. When the external magnetic field replacing stress is used to induce martensitic transformation the same regular should operate. If the temperature range of the martensitic transformation is too wide, only part of the martensitic transformation occurs, under this condition if the complete martensitic is required higher

Table 1 Martensitic transformation temperatures and Curie points of alloys

Alloy	Annealing temperature/K	Martensitic transformation temperature/K				ΔT_m/K	T_m/K	T_c /K
		M_s	M_f	A_s	A_f			
$Co_{41}Ni_{32}Al_{27}$	1 423			90*	139*	47*	129*	265
	1 473			141*	190*	49*	180*	301
	1 523	219	180	188	230	40	224.5	332
	1 573	232	194	229	270	39.5	251	375
	1 623	302	248	272	327	54.5	314.5	414
$Co_{41}Ni_{32}Al_{24}Sb_3$	1 423	190	168	200	226	24	208	284
	1 473	239	216	249	271	22.5	255	328
	1 523	293	271	297	320	22.5	306.5	364
	1 573	307	280	304	335	29	321.7	392

* Valuated from M—T curves; $\Delta T_m=(M_s-M_f+A_f-A_s)/2$; $T_m=(A_s+M_s)/2$

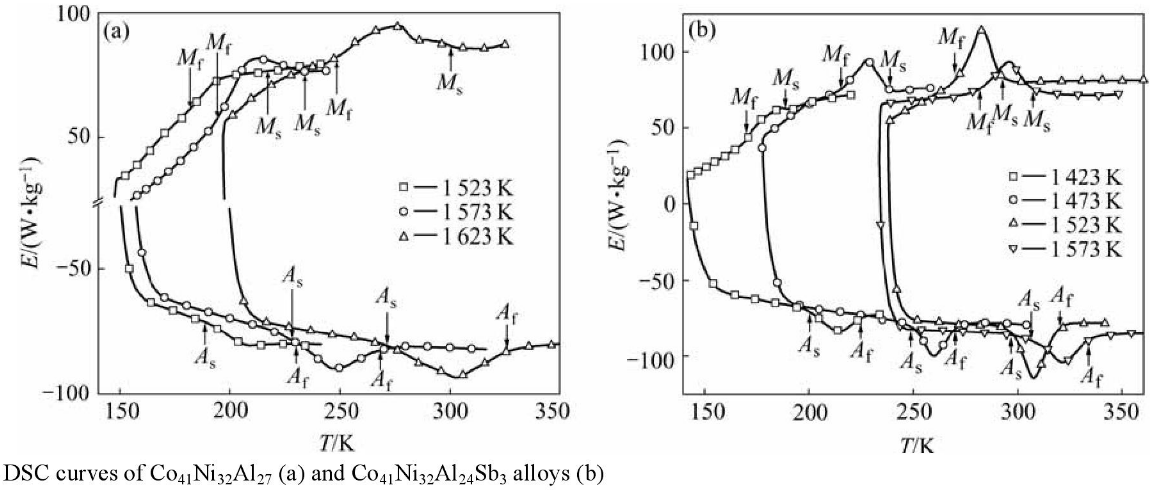

Fig.4 DSC curves of $Co_{41}Ni_{32}Al_{27}$ (a) and $Co_{41}Ni_{32}Al_{24}Sb_3$ alloys (b)

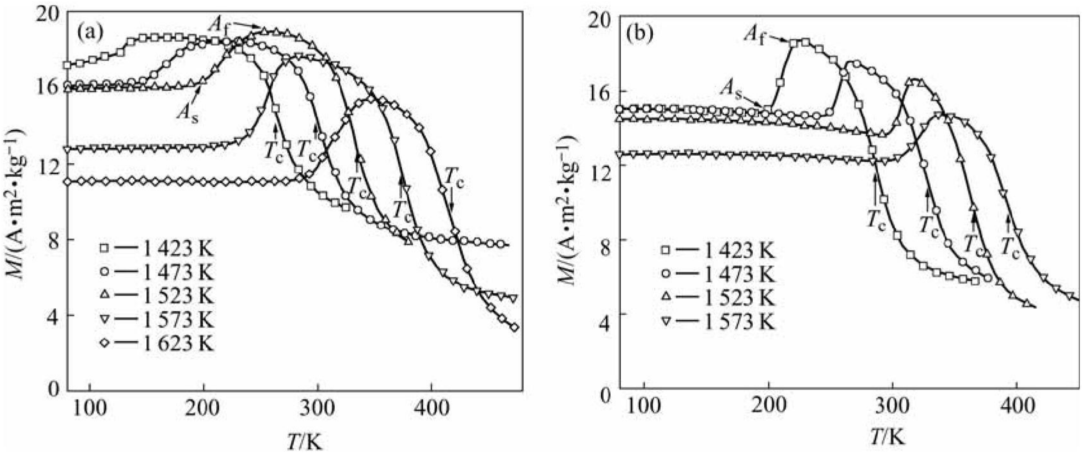

Fig.5 Thermomagnetization curves *(M—T)* of $Co_{41}Ni_{32}Al_{27}$ (a) and $Co_{41}Ni_{32}Al_{24}Sb_3$ alloys (b)

intense of magnetic field is needed. The intensity of the external magnetic field is difficult to improve and on the other side other physical phenomena, such as magneto contraction, may occur and so counteract the effect of the magneto phase transformation. In our research the martensite phase transformation temperature of the $Co_{41}Ni_{32}Al_{27}$ is 40−58 K and this is perhaps the main reason why the magneto strain of the Co-Ni-Al is only 0.06%[7].

$T_m=(A_s+M_s)/2$ is used to compare the T_m of the alloys and they are listed in Table 1. The relation between T_c/T_m and temperature is shown in Fig.6. It can be seen that both T_c and T_m are proportional to the quenching temperature and both the two T_m—T and the two T_c—T are parallel, which indicates that the changing rate of T_m & T_c with quenching temperature is uniform. T_m increases by 9 K and T_c increases by 18 K when the quenching temperature is enhanced by 10 K. Quenched at the same temperature, the T_m of the $Co_{41}Ni_{32}Al_{24}Sb_3$ is 76 K higher than that of the $Co_{41}Ni_{32}Al_{27}$ and T_c is 18 K higher. It is very important to improve the T_m and T_c of the Co-Ni-Al alloys for the development of the FSMA and it is favorable to get FSMA with high magneto strain[10,15]. The T_m and T_c of the $Co_{41}Ni_{32}Al_{24}Sb_3$ quenched at 1 573 K deviate from the rule mentioned above and this should be related to the part melting of $Co_{41}Ni_{32}Al_{24}Sb_3$, the initial melting temperature of which is marked by imaginary line in Fig.6.

4 Discussion

The change of the martensitic phase transform temperature could be explained by the change of average total s+d electron concentration *e/a*. In calculation the total s+d electron number of the Co, Ni, Al and Sb are relatively 9, 10, 3, 5. Because it is in *β* that the martensitic transformation occurs, namely only the *e/a* of the *β* is relative to T_m, so the chemical constitutions of the *β* was analyzed by EDX and the calculated *e/a* is show in Table 2. The T_m—*e/a* curve is shown in Fig.7. In general, the T_m is proportional to T_m[16,17], and it can be found from Fig.7 that the T_m of the two alloys studied in this paper accords with the rule.

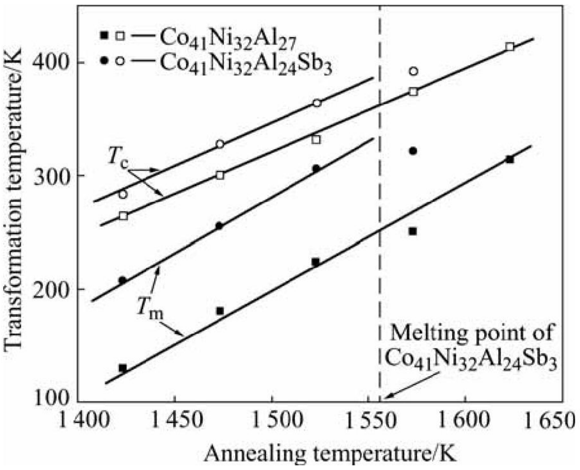

Fig.6 Relationship between T_m and T_c to annealing temperature

The relation between the T_m and the quenching temperature can be explained with the change of *e/a*. As shown in Fig.1, the volume fraction of the *γ* decreases with the increase of the quenching temperature. In Ni-Al alloys *γ* is a kind of fcc phase rich in Ni, and the Co in the $Co_{41}Ni_{32}Al_{27}$ and $Co_{41}Ni_{32}Al_{24}Sb_3$ takes the position of Ni in the Ni-Al alloys. Because the chemical constitutions do not change with the quenching temperature, the decrease of the *γ* which is rich in Co and Ni will increase the Co and Ni in the *β*. It can be found from Table 2 that in the two alloys the Co content monotone increases and the Al monotone decreases. Because the s+d electron number of Co is higher than that of Al which makes the *e/a* and T_m increase.

At the same quenching temperature, the phenomenon that the T_m of $Co_{41}Ni_{32}Al_{24}Sb_3$ is 76 K than that of $Co_{41}Ni_{32}Al_{27}$ can also be explained using *e/a*. On the one hand, the s+d electron number of Sb is higher

Table 2 Chemical composition, total average s+d electron concentration and magnetic valence number of β phase

Alloy	Annealing temperature/K	Chemical composition/%				e/a	Z_m
		Co	Ni	Al	Sb		
$Co_{41}Ni_{32}Al_{27}$	1 423	36.62	32.17	31.21		7.449	−0.570
	1 473	37.58	31.62	30.80		7.468	−0.548
	1 523	38.02	31.48	30.50		7.485	−0.535
	1 573	38.84	31.43	29.73		7.531	−0.504
	1 623	39.77	31.37	28.85		7.582	−0.468
$Co_{41}Ni_{32}Al_{24}Sb_3$	1 423	37.65	31.64	29.12	1.59	7.506	−0.577
	1 473	39.18	31.08	28.4	1.33	7.553	−0.527
	1 523	39.30	31.32	28.17	1.10	7.569	−0.507
	1 573	39.86	31.07	28.13	0.94	7.585	−0.492

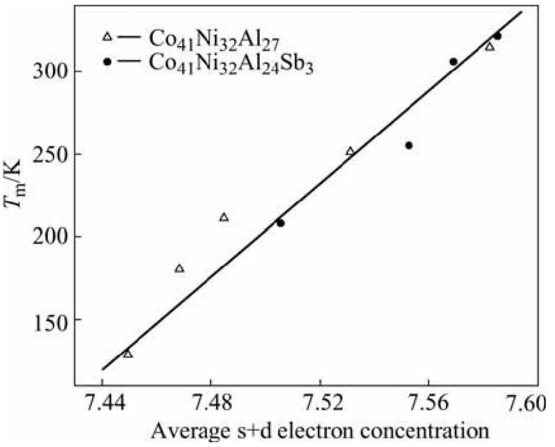

Fig.7 Martensitic transformation temperature T_m as a function of total average s+d electron concentration

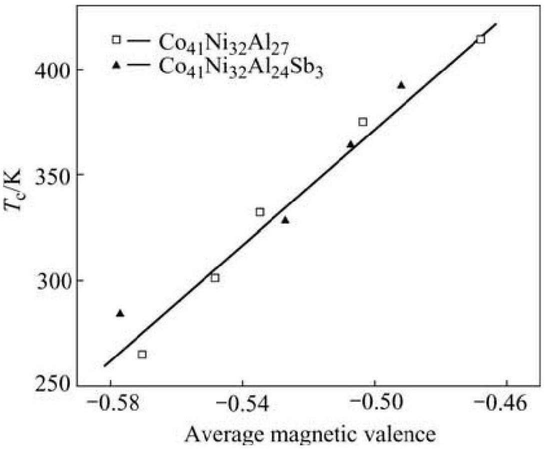

Fig.8 Curie point T_c as a function of average magnetic valence number Z_m

than that of Al and the substitution of Al with Sb will make the e/a increase; on the other hand comparing Fig.1(a) with Fig.1(e), at the same quenching temperature of 1 523 K, the volume fraction of γ in $Co_{41}Ni_{32}Al_{24}Sb_3$ is higher than that of the $Co_{41}Ni_{32}Al_{27}$, and this also serves to the increase of Co in β. It also can be seen from Table 2 that at the same quenching temperature, the Co content of β in $Co_{41}Ni_{32}Al_{24}Sb_3$ is higher than that in $Co_{41}Ni_{32}Al_{27}$, and the total content of the Al and Sb is lower than that in $Co_{41}Ni_{32}Al_{27}$. All these serve to the higher e/a in $Co_{41}Ni_{32}Al_{24}Sb_3$ and the increasing of T_m.

The T_c is decided by the chemical composition and the degree of order of the phase[16], while the degree of order is mainly decided by crystal structure. As shown in Fig.6, the T_c of the alloys is larger than T_m and they are in B2 structure of the parent phase when the magnetic transformation occurs, namely the degree of order of all the alloys is similar, and so the T_c is decided mainly by the chemical position of the β. In some last research, it is shown that T_c is related to magnetic valence electron number Z_m[17]. The Z_m of Co, Ni, Al, Sb is relatively 1, 0, −3, −5, and the Z_m of β of each alloy is listed in Table 2. The T_c—Z_m plot is shown in Fig.8 and it can be seen that T_c is proportional to Z_m.

The relation between T_c and the quenching temperature can be explained using Z_m. It can be seen from Table 2 that the content of Co increases and that of Al decreases. Because the Z_m of the Co and Al is 1 and −3, the increase of Co and the decrease of Al will result in the increase of Z_m and T_c.

T_c of $Co_{41}N_{i32}Al_{24}Sb_3$ alloy is higher than that of $Co_{41}Ni_{32}Al_{27}$ by 18 K when temperature of quench is the same. It can not be explained by Z_m of Sb, because Z_m of Sb is −5 and lower than Z_m of Al. If contents of Co and Ni in β phase is invariable, only part Al is substituted with Sb, it will lead to decrease of average Z_m. The result brings out decrease of T_c. But it is shown in Fig.1(a) and Fig.1(e) that, when the quenching temperature is the same as 1 523 K the volume fraction of γ phase in $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy is lower than that of $Co_{41}Ni_{32}Al_{27}$ alloy, which leads to Co increase in β phase. It can be seen from Table 2 that, when the quenching temperature is the same also, the content of Co is higher in β phase of $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy than $Co_{41}Ni_{32}Al_{27}$ alloy, but the total contents of Al and Sb are lower than that of $Co_{41}Ni_{32}Al_{27}$. T_c is increased since Z_m of $Co_{41}Ni_{32}Al_{24}Sb_3$ alloy is high.

5 Conclusions

1) In the $Co_{41}Ni_{32}Al_{24}Sb_3$ alloys, although the $L1_0$ type martensite still forms, the temperature range of the martensitic transformation is only 22−29 K, which is half that of the $Co_{41}Ni_{32}Al_{27}$ (40−55 K).

2) In the $Co_{41}Ni_{32}Al_{24}Sb_3$ alloys as well as in the $Co_{41}Ni_{32}Al_{27}$ alloys, T_m and T_c are both in proportion to the quenching temperature. T_m increases by 9 K and T_c increases by 7.5 K when the quenching temperature increases by 10 K.

3) When quenched at the same temperature, the T_m of the $Co_{41}Ni_{32}Al_{24}Sb_3$ is 76 K higher than that of the $Co_{41}Ni_{32}Al_{27}$ and the T_c is 18K higher.

4) In $Co_{41}Ni_{32}Al_{24}Sb_3$, eutectic structure appears when heated treatment at 1 573 K and the alloy is partly melted, while the $Co_{41}Ni_{32}Al_{27}$ alloys don’t still melt when heat treated at 1 623 K. So the addition of Sb to Co-Ni-Al alloy decreases the melting point.

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