Journal of Thermal Analysis, Vol. 38 (1992) 397-407

IS THE DSC TECHNIQUE CAPABLE OF DETERMINING ACCURATE SPECIFIC HEATS OF METALLIC RIBBONS?

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Using the scanning (differential) method and a PE DSC in continuous heating regime as well as the integral (enthalpic) method and a SETARAM DSC in a discontinuous heating regime the specific heats of several metallic glassy ribbons were measured. The linear $c_{pM}(T)$ dependencies in the as-quenched, relaxed and also the stable crystalline forms of the ribbons in the medium temperature region were found. The room temperature specific heats of these ribbons, the excess contributions due to the relaxed glassy state as well as the non-relaxed glassy state were determined.

The sensitivity of $c_{pM}(T)$ measurements of metallic ribbons was better than 0.2%, the long term reproducibility in the case of glassy ribbons was 2%. They are related to the errors in $c_{pM}(T)$ measurements of synthetic sapphire standard or bulk chalcogenide glasses.

Keywords: metallic ribbons, specific heat

Introduction

Metallic glasses represent a relatively new scientific area. In the medium temperature region they have heat capacities typical of metals but relatively low thermal conductivity. Some methodological problems concerning their calorimetry are connected with their ribbon form (the sample thickness of about 30 μm, mass of several tens of mg) and extremely ductile mechanical properties resulting in deformed edges of cut pieces of samples with problematic thermal contacts.

There are only very few specific heat data $c_p(T)$ at room or elevated temperatures of metals and practically none on glassy metallic ribbons. A review of experimental techniques and fundamental methodological steps of determination of specific heat $c_{pM}(T)$ of solids especially metals, alloys and glassy metallic alloys at elevated temperatures is given in Table 1.

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Table 1 Recent developments in experimental determination of specific heat $c_{pM}(T)$ of selected solids with relation to the glassy metallic alloys at elevated temperatures

Reference	Comment to the reference*	error
[1]	Adiabatic calorimeter, 300–550 K, Al_2O_3	±0.1%
[2]	Adiabatic calorimeter, 323–523 K, glassy $Ge_{20}Te_{80}$, as-quench., relaxed, cryst. state, struct. relaxation anomalies, ±1%	
[1]	Dynamic adiabatic calorimeter, till 1300 K, metals	±0.7%
[1]	Kubachevski adiabatic calorimeter, 700–1700 K, metals	±1%
[1]	Drop calorimeter, metals	±1.5%
[1]	Adiabatic drop calorimeter, 400–1400 K,$CuFeS_2$	±2%
[1]	Laser flash calorimeter, 80–1100 K, metals	±1%
[1]	Pulse differential calorimeter, till 3000 K, NbC, TiC	±3%
[4]	Subsecond pulse-heating method, 10^9 deg/s, 1000–2500 K, Nb	±3%
[1]	Direct pulse heating calorimeter, 300–1270 K, SiC	±0.5%
[5]	PE DSC, glassy ribbons, AuSi, undercooled liquid, T_g	±2%
[6]	PE DSC1, dividing 3 runs method, analysis of errors, zeolite	±1.5%
[7]	PE DSC2, methodic, scanning m., integral m., 3 runs, FeS_2	±1%
[8]	SETARAM DSC, integral stepwise m. theory, 300–1050 K, glassy ribbon $Fe_{80}B_{20}$ as quench., cryst. state, not interpreted	not giv.
[9]	PE DSC2, $F(T)$ correction factor, PVP	±1%
[10]	SETARAM DSC111, anharmonicity T^{-2}, $CuGe_2P_3$	±1%
[11]	PE DSC, form effects, quartz	not giv.
[12]	PE DSC2, subtraction 3 runs method, 300–800 K, glassy ribbon $Fe_{78}Si_{13}B_9$, as-quench. state, not interpreted	±5%
[13]	PE DSC2, glassy ribbon $Ni_{40}Pd_{40}P_{20}$, as-quench., cryst. state, interpreted abs. values	±5%
[14]	Mettler, SETARAM DSC, integral stepwise 3 runs m., glycerol	±3%
[15]	Power-compensated DSC, theory, 3 runs m., NaOH–$NaNO_3$	±3%

* characteristics of measuring technique, measuring temperature interval, measured material, methodological contribution,...

The technique of differential scanning calorimetry (DSC) has generally been regarded by the most classical calorimetrists as an untrustworthy means of acquiring accurate thermodynamic data. It was the intent of our previous work [16] to develop a correct scanning method and an integral method by means of a DSC instrument to measure accurate specific heats of metallic glassy ribbons. The present study gives an example of apparent specific heats of as-quenched, relaxed and crystalline metallic glasses $Fe_{73}Co_{12}B_{15}$, $Co_{67}Cr_7Fe_4Si_8B_{14}$ and $Co_{71.5}Mn_2Mo_1Fe_{2.5}Si_9B_{14}$ at temperatures from 300 to 1054 K.

Experimental

The $Fe_{73}Co_{12}B_{15}$, $Co_{67}Cr_7Fe_4Si_8B_{14}$ and $Co_{71.5}Mn_2Mo_1Fe_{2.5}Si_9B_{14}$ metallic glassy ribbons several meters long were prepared by the melt spinning technique with crossections 35 μm×10 mm, 25 μm×30 mm and 38 μm×30 mm always ±1.5 μm, respectively.

The technique of DSC was used to measure the temperature dependence of specific heat $c_{pM}(T)$ of ribbons. The power-compensated Perkin-Elmer DSC7 with PE 3700 DATA STATION as well as heat-flux SETARAM DSC111 with Commodore Data Station were used.

Four main principal representations of measured data were studied. Namely: (1) heat-power thermograms measured on PE DSC in the continuous heating regime, (2) heat-flux thermograms measured on SETARAM DSC in the continuous heating regime, (3) the specific heats measured and calculated by the scanning (differential) method using the PE DSC in a continuous heating regime and the PE DSC7 Specific Heat Software, (4) the specific heats measured and calculated by the integral (enthalpic) method using the SETARAM DSC in a discontinuous, so-called stepwise, heating regime and a special software [8].

The heating rates w used in measurements of type (1) and (2) were from 2 to 40 deg/min. The optimal heating rate was 5 deg/min in measurements of type (3) and $w = 5$ deg/min for 200 s followed by isothermal stabilization periods of 400 s (the quasi-linear mean heating rate $\overline{w} = 1.6$ deg/min) in discontinuous stepwise heating regime in experiments of type (4). The masses investigated in PE DSC were m~50 mg and in the case of SETARAM DSC m~360 mg. The samples were cut into small pieces. The reference was an empty Al pan in PE DSC and an empty Al_2O_3 pan in the case of SETARAM DSC. All measurements were done in an argon atmosphere.

Principles of specific heat measurements by DSC technique

Assuming a simple model, the fundamental differential or integral calorimetric equations describing the heat transfer in DSC experiment are written as follows:

$$\frac{\mathrm{d}H_{\mathrm{i}}(t)}{\mathrm{d}t} + C_{\pi}\cdot\frac{\mathrm{d}T_{\mathrm{E}}}{\mathrm{d}t} = \frac{1}{K_{\mathrm{i}}(t)}\cdot\left[Y_{\mathrm{i}} + \tau_{\mathrm{i}}\cdot\frac{\mathrm{d}Y_{\mathrm{i}}}{\mathrm{d}t}\right] - J_{\mathrm{i}}' \tag{1}$$

$$\int_{t1}^{t2}\frac{\mathrm{d}H_{\mathrm{i}}(t)}{\mathrm{d}t} + \frac{\mathrm{d}T_{\mathrm{E}}}{\mathrm{d}t}\cdot\int_{t1}^{t2} C_{\pi}\mathrm{d}t = \int_{t1}^{t2}\frac{Y_{\mathrm{i}}}{K_{\mathrm{i}}(t)}\mathrm{d}t + \int_{t1}^{t2}\frac{\tau_{\mathrm{i}}}{K_{\mathrm{i}}(t)}\cdot\frac{\mathrm{d}Y_{\mathrm{i}}}{\mathrm{d}t}\mathrm{d}t - \int_{t1}^{t2} J_{\mathrm{i}}'\mathrm{d}t \tag{2}$$

where i refers respectively to the measured sample $i = X$ and the reference sample $i = R$, $\mathrm{d}H_{\mathrm{i}}/\mathrm{d}t$ is the heat generation rate due to the eventual heat generation inside X, C_{π} is the heat capacity of the sample pan together with the sample placed inside it, $(C_{\mathrm{pPX}} + C_{\mathrm{pX}})$ or $(C_{\mathrm{pPR}} + C_{\mathrm{pR}})$, $1/K_{\mathrm{i}}(t)$ and Y_{i} are the sensitivity and the signal registered by one of the two sensors of the DSC detecting heads, respectively, τ_{i} is the time constant of the pan (with the sample) and J_{i}' is the heat flux to the surroundings, $w = \mathrm{d}T_{\mathrm{E}}/\mathrm{d}t$ is the heating rate driven by the DSC programmer to be constant.

(1) In a power-compensated DSC the registered electrical power $\Delta J_{(\mathrm{X-R})}$ is proportional to the difference of heat capacities of measured and reference sample pans with samples (Eq. (3) in [16])

$$\Delta J_{(\mathrm{X-R})}(T) \doteq C_{\mathrm{pPX}}(T) - C_{\mathrm{pPR}}(T) - C_{\mathrm{pR}}(T) \tag{3}$$

(2) In a heat-flux DSC the registered temperature difference $\Delta T_{(\mathrm{X-R})}$ is related to the heat capacities and thermal contact resistances $R(T)$ between heaters and measured pans (Eq. (5) in [16])

$$\frac{\Delta T_{(\mathrm{X-R})}(T)}{R(T,t)} \doteq C_{\mathrm{pPX}}(T) + C_{\mathrm{pX}}(T) - C_{\mathrm{pPR}}(T) - C_{\mathrm{pR}}(T) \tag{4}$$

$R(T, t)$ is a principally unknown temperature dependent proportionality coefficient of a heat-flux DSC instrument.

(3) and (4) The principles of the scanning and integral methods of measurement of absolute values of $c_{\mathrm{pM}}(T)$ of metallic ribbons are described and analyzed in [16]. The references used were always empty pans R = Blanc, $C_{\mathrm{pR}} = 0$, C_{pPR} = const. Both methods consist of two measuring cycles: one with an unknown sample $X = M$ and the equivalent one with a standard which in our case was an empty unknown sample pan $X = S$ = Blanc, $C_{\mathrm{pS}} = 0$, $C_{\mathrm{pPS}} = C_{\mathrm{pPM}}$. In the scanning method the measured $c_{\mathrm{pM}}(T)$ at any temperature is proportional to the subtraction of corresponding measured electrical powers (Eq. (7) in [16])

$$c_{\mathrm{pM}}(T) \doteq \frac{\Delta J_{(\mathrm{M-R})}(T) - \Delta J_{(\mathrm{S-R})}(T)}{w \cdot m_{\mathrm{M}}} \tag{5}$$

assuming $\mathrm{d}H_{\mathrm{M}}(T)/\mathrm{d}t = 0$, neglecting the minor differential terms $\mathrm{d}Y_{\mathrm{i}}(T)$, m_{M} being the mass of M. In the integral method the mean $\overline{c_{\mathrm{pM}}(T)}$ is proportional to the difference of areas below the measured signals during a short temperature interval (Eq. (8) in [16])

$$\overline{c_{\mathrm{pm}}(T_1, T_2)} = $$

$$= \frac{\frac{1}{w}\left[\int\limits_{T_1}^{T_2} \Delta T_{(\mathrm{M-R})}\mathrm{d}T - \int\limits_{T_1}^{T_2} \Delta T_{(\mathrm{S-R})}\mathrm{d}T\right] + \tau\left\{\int\limits_{1}^{2}\mathrm{d}\left[\Delta T_{(\mathrm{M-R})}\right] - \int\limits_{1}^{2}\mathrm{d}\left[\Delta T_{(\mathrm{S-R})}\right]\right\}}{m_{\mathrm{M}} \cdot (T_2 - T_1) \cdot \overline{K(T_1, T_2)}} \tag{6}$$

assuming $\mathrm{d}H_{\mathrm{M}}(T) = 0$. The mean sensitivity coefficient $\overline{K(T_1, T_2)}$ is being calibrated by an equivalent measuring cycle experiment on some known sample M.

The correctness of the theoretical principles and the accuracies of obtained experimental data on metallic ribbons from 300 to 1054 K were analyzed in [16]. These are summarized in Table 2.

Results and discussion

Metallic glasses are not stable from the thermodynamic point of view. That's why the glassy metallic ribbons need to be measured always in 3 successive heating runs: 1^{st} run characterizing the as-quenched glassy state, 2^{nd} run characterizing the ideally relaxed state and 3^{rd} run characterizing the final stable crystalline state of a sample.

Examples of typical continuous heating regime heat-power or heat-flux thermograms measured by the measuring techniques (1) and (2) are in Figs 1a and 1b. The complex behaviour of such representations of $c_{\mathrm{pM}}(T)$ having one pronounced structural relaxation exotherm and some possible anneal induced endotherms followed by several crystallization exotherms have been studied in [17]. In these cases the measured signals $\Delta J_{(\mathrm{M-R})}(T)$ and $T_{(\mathrm{M-R})}$ are just proportional to the $c_{\mathrm{pM}}(T)$ of measured samples and their absolute values are only relative.

Table 2 Statistical errors of measuring specific heat of metallic ribbons by the use of SETARAM DSC111 instrument and the integral method and of Perkin–Elmer DSC7 instrument and the scanning method

	SETARAM DSC111				Perkin–Elmer DSC7			
	at 300 K		at 1054 K		at 300 K		at 468 K	
inaccuracy in T / K	±1.		±1.		±0.5		±0.5	
inaccuracy in $c_{p(Co)}$ /Jg^{-1} at K	±0.7	±2.8%	±0.5	±1.4%	–1.9	–7.7%	–2.2	–7.6%
sensitivity of T / K	±8.		±8.		±0.01		±0.01	
sensitivity of $c_{p(FeCoB)}$ / Jg^{-1}		±0.01	±0.04%			±0.05	±0.2%	
max. noise of $c_{p(Co)}$ /Jg^{-1} at K	±0.2	±0.8%	±0.4	±1.0%	±0.05	±0.2%	±0.05	±0.2%
max. dispersion of $c_{p(FeCoB)}$ / Jg^{-1} at K	±0.2	±0.8%	±0.4	±1.8%			±0.24	±1.2%
reproducibility of $c_{p(Co)}$ / Jg^{-1}					±0.08	±0.4%	±0.04	±0.2%
reprod. of $c_{p(FeCoB,\ as\text{-}q)}$ / Jg^{-1}	±0.6	±2.5%			±0.48	±2.0%	±0.61	±2.3%
reprod. of $c_{p(FeCoB,\ cryst)}$ / Jg^{-1}	±0.4	±1.8%	±0.4	±1.1%	±0.38	±1.9%	±0.41	±1.6%
reprod. of $c_{p(CoCrFeSiB,\ cryst)}$ / Jg^{-}					±0.05	±0.3%	±0.02	±0.2%
inaccuracy in $c_{p(Al_2O_3)}$ /Jg^{-1} at K*					±0.30	±1.9%	±0.36	±1.8%
reprod. of $c_{p(Al_2O_3)}$ /Jg^{-1} at K*					±0.06	±0.4%	±0.12	±0.6%
reprod. of $c_{p(SeTeGe,as-q)}$ /Jg^{-1} at K*					±0.71	±2.8%	±0.1	±0.3%

* done on Perkin–Elmer DSC2 instrument

The absolute values of $c_{pM}(T)$ of studied materials measured by both measuring techniques (3) and (4) are in Figs 1c, 1d, 2a and 2b.

The major contributions to the medium temperature heat capacities $C_p(T)$ of metallic ribbons are the harmonic vibrational heat capacity $C_v \sim 3R$,

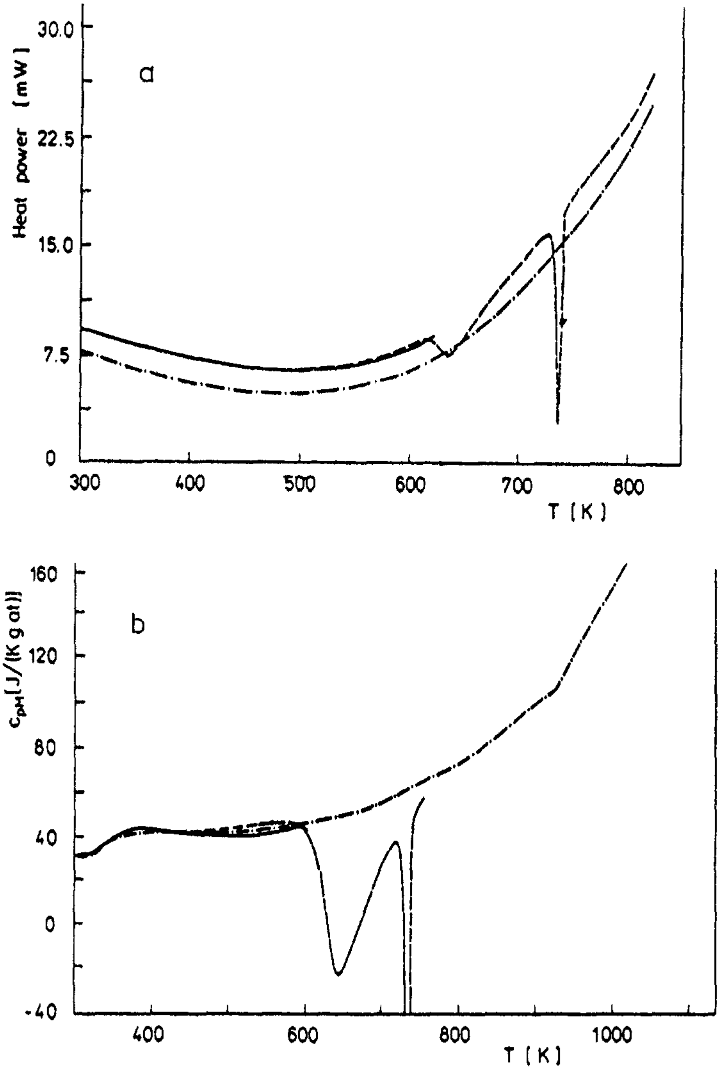

Fig. 1a,b Temperature dependencies of specific heat of $Fe_{73}Co_{12}B_{15}$ ribbon in the as-quenched glassy (———), relaxed glassy (— — —) and crystalline (— · — · —) forms: 1a – the PE DSC thermograms (w = 40 deg/min), 1b – the SETARAM DSC thermograms (w = 2 deg/min) the quasi-harmonic dilation heat capacity $C_d(T)$ $\sim\beta^2VT/\chi$ as well as the explicit anharmonic excess contributions to $C_v(T)$. Then the absolute values as well as the slopes of the $c_{pM}(T)$ dependencies are related to the lattice vibrational spectra, the specific volume V, expansivity β, compressivity χ and degree of thermodynamical non-equilibrium of relaxing samples. As can be expected from previous studies of metallic glasses [5, 12, 13] room temperature apparent (measured) specific heats of as-quenched, relaxed and crystalline metallic ribbons have similar absolute values (Table 3). These results correlate well with fact of existence of some short range ordering in the glassy structure which is related to the crystalline one.

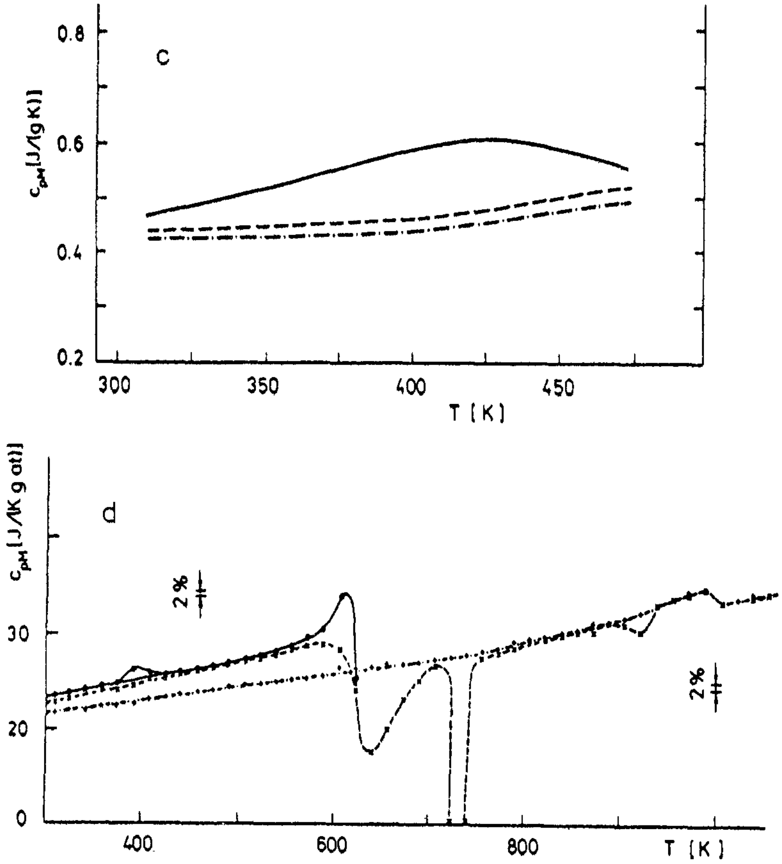

Fig. 1c,d Temperature dependencies of specific heat of $Fe_{73}Co_{12}B_{15}$ ribbon in the as-quenched glassy (———), relaxed glassy (— — —) and crystalline (— · — · —) forms: 1c – measured and calculated by PE DSC and scanning method (w =5 deg/min), 1d – measured and calculated by SETARAM DSC and integral method ($\overline{w}$ =1.6 deg/min)

If in the sample some transformation occurs the total $c_{pM}(T)$ evolves according to the changing amount of initial and final phases c_{p1} and c_{p2} as well as the excess heat capacity due to the thermal effect in the sample appears

$$c_{pM}\left(T,t\right) = \left[1-\alpha\left(T,t\right)\right]c_{p1}\left(T\right) + \alpha\left(T,t\right)c_{p2}\left(T\right) + \Delta H_M \frac{d\alpha\left(T,t\right)}{dt}\cdot\frac{1}{m_M} \qquad (7)$$

where α is the degree of transformation fulfilling a reasonable kinetic equation and ΔH_M is the enthalpy change of the transformation.

But there are two principal problems which do not allow the exact interpretation of eventual calculated kinetic parameters: (a) The initial-

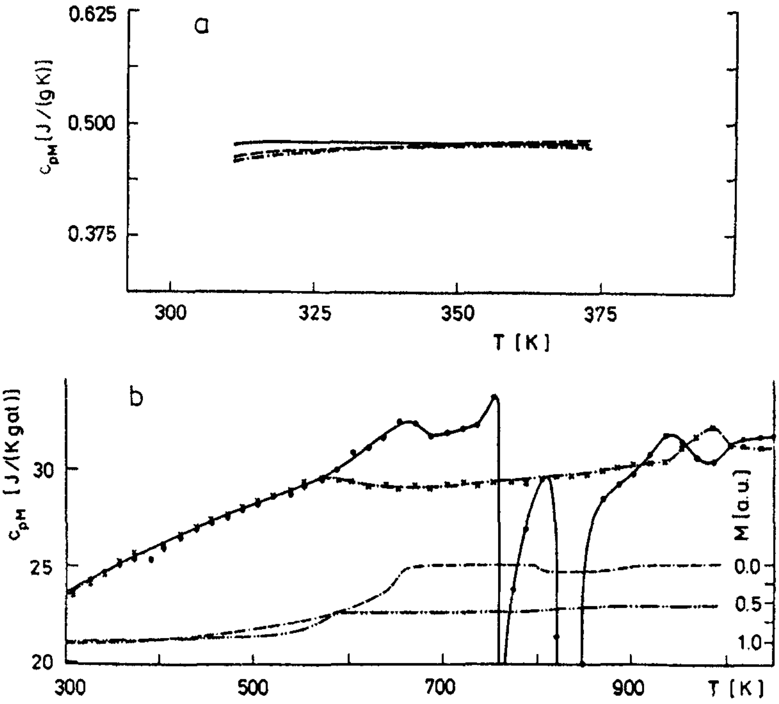

Fig. 2 Temperature dependencies of specific heat $c_{pM}(T)$ of as-quenched glassy (———), relaxed glassy (— — —) and crystalline (— · — · —) forms: 2a – $Co_{67}Cr_7Fe_4Si_8B_{14}$ ribbon measured by PE DSC and scanning method (w = 5 deg/min), 2b – $Co_{71.5}Mn_2Mo_1Fe_{2.5}Si_9B_{14}$ ribbon measured by SETARAM DSC and integral method ($\bar{w}$ = 1.6 deg/min). Curves (— — · — — · — —) and (— ·· — · · —) are temperature dependencies of magnetization M(T) of as-quenched glassy and crystalline samples, respectively

$c_{\mathrm{p1}}=c_{\mathrm{p(glass)}}(T, t)$ is not well defined because of the thermodynamic instability, e.g. structural relaxation of the glassy state of the sample [17], having the enthalpy $\Delta H_{\mathrm{relax}} < 0.1\ \Delta H_{\mathrm{cr1}}$. (b) The correct theoretical kinetic equation for any transformation being a multimechanism process in real solids, in metallic glasses especially, does not exist yet because of the unknown spectrum and time and temperature dependence of transformation 'constant' $k(T, t) = \sum_{i} g_i k_i(T, t)$ or probably $k(T, t) = \sum_{i} g_i(T, t)\, k_i(T)$.

On the other side, in the case of method (4) the kinetic information of the mean values $\overline{c_{\mathrm{pM}}(T_1, T_2)}$ is only schematic.

Table 3 Mean values of specific heat c_{pM} (323 K) in $\mathrm{Jg^{-1}}$ at K of several metallic ribbons in as-quenched, relaxed and crystalline states

chem. composition	$c_{\mathrm{pM(as\text{-}q)}}$	$c_{\mathrm{pM(r)}}$	$c_{\mathrm{pM(cr)}}$	$c_{\mathrm{pM(as\text{-}q.\text{-}r.)}}$	$c_{\mathrm{pM(as\text{-}q.\text{-}cr.)}}$
$Fe_{73}Co_{12}B_{15}$	24.72	23.87	22.50	+0.85	+2.22
$Co_{67}Cr_7Fe_4Si_8B_{14}$	23.46	22.96	22.91	+0.50	+0.55
$Co_{71.5}Mn_2Mo_1Fe_{2.5}Si_9B_{14}$	24.37		24.10		+0.27

By the help of the electron microscopy phase analysis of crystallization of metallic glasses the $c_{\mathrm{pM}}(T)$ crystallization exotherms can be identified e.g. as in the case of $Fe_{73}Co_{12}B_{15}$ ribbon the polymorphous crystallization of supersaturated α-Fe glassy regions above 600 K followed by the α–Fe crystallization of the remaining amorphous regions absorbing the parts crystallized before together with precipitation of Fe_3B above 700 K and finished by the recrystallization to Fe_2B above 900 K. By the help of measuring magnetization, e.g. in the case of $Co_{71.5}Mn_2Mo_1Fe_{2.5}Si_9B_{14}$ ribbon the Curie point of glassy material at about 660 K, the Curie point of some of the crystalline phases above 900 K (or of the crystalline α-Fe in the case of $Fe_{73}Co_{12}B_{15}$ ribbon at about 1000 K) or some reversible structural change of the crystalline Co-rich material losing its feromagnetic state can be identified.

Conclusion

Both kinds of DSC calorimeters: power-compensated Perkin-Elmer DSC7 and heat-flux SETARAM DSC111 have been tested. The sensitivity of $c_{\mathrm{pM}}(T)$ measurements of metallic ribbons was better than 0.2%, the long term reproducibility in the case of glassy ribbons was 2%. The specific heats $c_{\mathrm{pM}}(T)$ of $Fe_{73}Co_{12}B_{15}$, $Co_{67}Cr_7Fe_4Si_8B_{14}$ and $Co_{71.5}Mn_2Mo_1Fe_{2.5}Si_9B_{14}$ glassy ribbons in as-quenched, relaxed and crystalline states in the medium temperature range were measured.

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Zusammenfassung — Unter Anwendung des Scanning- (Differential-) Verfahrens und einem PE DSC in kontinuierlichen Heizsystemen sowie des Integral- (Enthalpie-) Verfahrens und einem SETARAM DSC in diskontinuierlichen Heizsystemen wurde die spezifische Wärme diverser metallisch-glasartiger Bänder gemessen. Im abgeschreckten, entspannten Zustand und auch in den kristallinen Formen der Bänder konnte in der mittleren Temperaturregion eine lineare $c_{pM}(T)$ Abhängigkeit gefunden werden. Sowohl die spezifischen Wärmen dieser Bänder bei Raumtemperatur, der zusätzliche Beitrag im entspannten glasartigen Zustand als auch im nichtentspannten glasartigen Zustand wurden bestimmt.
Die Empfindlichkeit bei $c_{pM}(T)$-Messungen metallischer Bänder war besser als 0.2%, die Langzeitreproduzierbarkeit betrug im Falle glasartiger Bänder 2%. Sie wurden auf die Fehlergrößen von $c_{pM}(T)$-Messungen an einem synthetischen Saphir-Standard oder an massiven Chalkogenidgläsern bezogen.