Thermophysical Properties of Platinum Group Metals in their Liquid Undercooled and Superheated Phases

Measurements in the liquid state, including the undercooled region, over wide temperature ranges with an electrostatic levitator

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This review briefly describes the vacuum electrostatic levitation furnace developed by JAXA and the associated non-contact techniques used to measure the density, the surface tension and the viscosity of materials. The paper then presents a summary of the data taken with this facility in the equilibrium liquid and non-equilibrium liquid phases for the six platinum group metals (pgms): platinum, palladium, rhodium, iridium, ruthenium and osmium over wide temperature ranges that include undercooled and superheated phases. The presented data (density, surface tension and viscosity of Pt, Rh, Ir, Ru and Os and density of Pd) are compared with literature values.

1. Introduction

Due to their unique characteristics such as inertness, corrosion and oxidation-resistance, biocompatibility, catalytic behaviour, high melting temperatures and good conductivity, the pgms are used in a host of applications (for example, automotive, aerospace, electronics, industrial heating, medical and jewellery) (1). To design high-performance alloys and to optimise industrial processes (for example, refining, casting and welding), knowledge of the density, surface tension and viscosity and their temperature dependences is often required. This understanding is required not only for the equilibrium liquid phase but also for the non-equilibrium, undercooled phases, because alloys with new microscopic structures can be synthesised from such phases. However, the high melting temperature of pgms (Pd: 1828 K; Pt: 2041 K; Rh: 2236 K; Ru: 2607 K; Ir: 2720 K; Os: 3306 K) (1) and the risk of contamination at elevated temperatures make measurements of their thermophysical properties of their equilibrium and non-equilibrium liquid phases very challenging using traditional methods.

The electrostatic levitation furnace and the associated non-contact diagnostics techniques developed by JAXA over the years (2, 3) have circumvented the difficulties associated with high-temperature processing and allowed an accurate determination of several properties of the pgms (4–13). High-temperature processing was achieved in vacuum using laser heating, thus isolating the sample from contaminating walls as well as surrounding gases. The containerless processing conditions also permit a deep undercooling of the material samples because of minimised heterogeneous nucleation and because sample heating and levitation were independent.

This paper first briefly describes the facility and the property measurement methods and then summarises

the experimental data for the density, surface tension and viscosity for the liquid phase of the pgms.

2. Experimental

2.1 Electrostatic Levitator

Properties were measured using an improved electrostatic levitator (**Figure 1**) (2, 3) in a vacuum environment (~10^{-5} Pa) originally based on a design by Rhim *et al.* (14). With this instrument, *ca.* 2 mm diameter samples charged by electronic emission were levitated one at a time between electrodes. A feedback loop between the applied electric fields and the position information obtained from the shadow of the sample illuminated with helium-neon lasers and detected by sensors ensured stable levitation. The sample was heated with up to four focused laser beams: three carbon dioxide laser beams (10.6 µm, total power 200 W) separated by 120º in a horizontal plane and one neodymium-doped yttrium aluminium garnet (Nd:YAG) laser beam (1.064 µm, 500 W) from the top. A complete description of the facility and the levitation initiation procedures can be found elsewhere (2, 3, 10, 12). This configuration provided temperature homogeneity and sample position stability and allowed control of sample rotation. The radiance temperature was measured with a single-colour pyrometer (0.90 µm, 120 Hz acquisition rate). The sample was observed by black and white high-resolution charged-coupled-device cameras. The cameras were located at right angles from each other and were equipped with telephoto lenses in conjunction with background illumination lamps. This provided magnified views of the sample and helped to monitor the sample position and to align the heating laser beams.

2.2 Property Measurements

This levitation furnace was particularly suitable to measure the density, surface tension and viscosity of pgms in their equilibrium and undercooled liquid phases. Since sample heating and levitation were independent, a precise laser heating control allowed undercooled melts to be maintained for time scales much longer than those required for the measurements.

2.2.1 Density

Density measurements were carried out using an imaging technique described elsewhere (3, 15). Once a levitated sample was melted, it took a spherical shape due to surface tension and the distribution of surface charge and images from a high-resolution camera (30 frames per second) and temperature data were simultaneously recorded with time (**Figure 2**). The laser beams were then blocked with mechanical shutters allowing the sample to cool radiatively. After the experiment, each image was matched with the
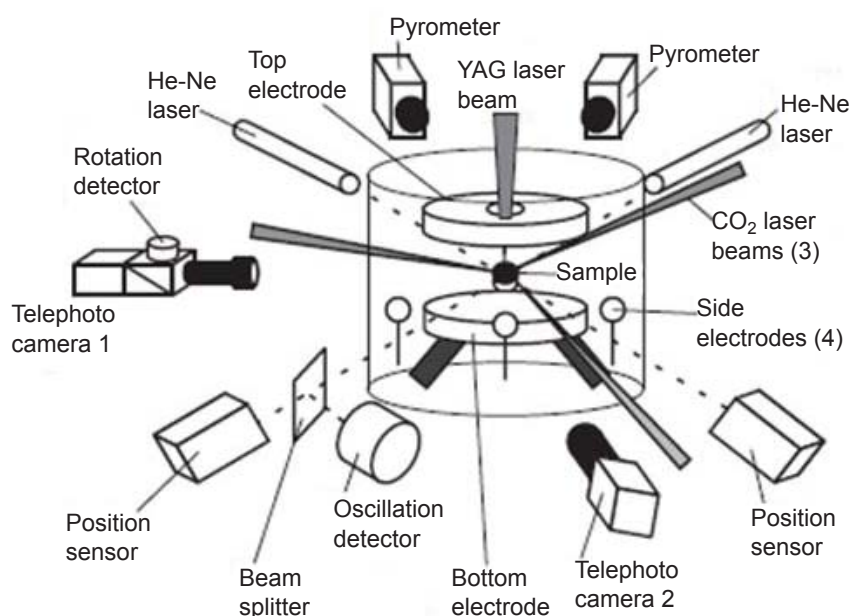

Fig. 1. Schematic view of the electrostatic levitation furnace

Fig. 2. Representative image of a levitated molten Os sample (~2 mm diameter)

thermal history of the sample (**Figure 3**), digitised and the sample radius was extracted by software. Since the sample was axi-symmetric and because its mass was known, the density was found as a function of temperature. Calibration was done by levitating a sphere with a precisely known radius under identical experimental conditions.

2.2.2 Surface Tension and Viscosity

The surface tension was determined by the oscillation drop technique, a method in which the frequency of the oscillation of a levitated molten sample about its equilibrium shape is measured (16). This technique was explained earlier (17) and is summarised below for completeness. In this method, a sample was first heated, melted and brought to a selected temperature, while ensuring excellent position stability, low sample rotation and sample sphericity. Then, a $P_2 \cos(\theta)$-mode drop oscillation was induced to the sample by superimposing a small sinusoidal electric field on the levitation field. Following the termination of the excitation field, the transient signal generated by the change in diameter of the oscillating drop was detected and analysed using a custom made program. This was done many times for a given temperature and repeated for numerous temperatures. Using the characteristic oscillation frequency ω_c of this signal, which was calculated by a fast Fourier transform and then corrected for

nonuniform surface charge distribution (18), the surface tension σ could be found from Equation (i) (17, 19):

$$\omega_c^2 = (8\sigma/r_o^3\rho)\,[1 - (Q^2/64\pi^2 r_o^3\sigma\varepsilon_o)][1 - F(\sigma,q,e)] \quad \text{(i)}$$

where

$$F(\sigma,q,e) = [243.31\sigma^2 - 63.14q^2\sigma + 1.54q^4]e^2\,/$$
$$[176\sigma^3 - 120q^2\sigma^2 + 27\sigma q^4 - 2q^6] \quad \text{(ii)}$$

and r_o is the radius of the sample when it assumed a spherical shape, ρ is the liquid density, Q is the drop charge, ε_o is the permittivity of vacuum and q and e are respectively defined by Equations (iii) and (iv):

$$q^2 = Q^2/16\,\pi^2 r_o^3\varepsilon_o \quad \text{(iii)}$$

and

$$e^2 = E^2 r_o\,\varepsilon_o \quad \text{(iv)}$$

with E being the applied electric field (levitation and excitation). Similarly (17, 20), using the decay time τ given by the same signal, the viscosity η was found by Equation (v):

$$\eta = \rho r_o^2/(5\tau) \quad \text{(v)}$$

In Equations (i) and (v), the value of the density ρ at the corresponding temperature and the real-time value of the radius r_o, obtained by the imaging technique, were used so that sample evaporation does not distort the surface tension and the viscosity data.

3. Experimental Results
3.1 Density

All pgm samples were successfully levitated, melted, undercooled and solidified with the electrostatic levitator (4–8, 10, 11) and the processing data specific for each metal are listed in **Table I**. During these experiments, the density of equilibrium and non-equilibrium liquid phases was measured over large temperature ranges that cover the superheated and undercooled regions. **Figure 4** presents typical plots for all pgms. The data measured by electrostatic levitation (4–6, 8, 10, 11) together with the literature values (21–36) are summarised in **Table II**. To our knowledge, these levitation measurements are the only ones that explore the undercooled region to date. The density of the pgms, like that of other pure metals, exhibited a linear behaviour as a function of temperature. In the density measurements, the uncertainty was estimated to be less than 2% from the resolution of the video grabbing
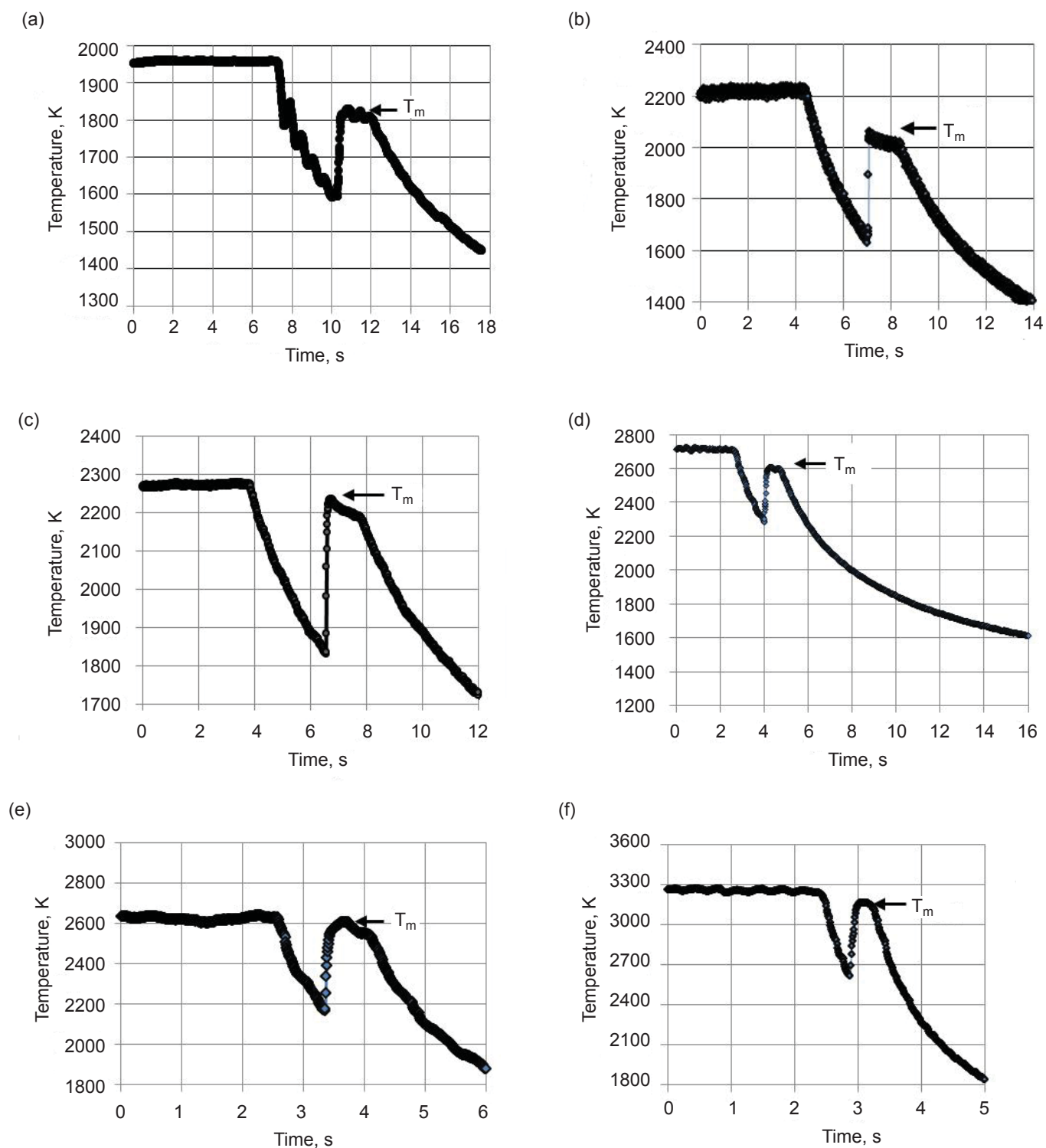
Fig. 3. Representative thermal history obtained for millimetre size pgm samples showing heating above the melting temperature, radiative cooling, undercooling, recalescence and solidification: (a) Pd (6); (b) Pt (11); (c) Rh (4); (d) Ru (5); (e) Ir (8); and (f) Os (10). (T_m = melting temperature)

Table I Processing Data for Platinum Group Metals (4–6, 8, 10, 11)				
Metal	**Melting temperature, K**	**Purity, wt%**	**Undercooling, K**	**Superheating, K**
Pd	1828	99.95	188	47
Pt	2041	99.8	340	170
Rh	2236	99.9	416	14
Ru	2607	99.9	382	168
Ir	2720	99.9	420	280
Os	3306	99.9	575	35

capability (640 × 480 pixels) and from the uncertainty in mass (±0.0001 g).

At the melting point, the values obtained by levitation agree generally very well with those measured by other techniques (for example, the drop weight technique, isobaric expansion, pendant drop and sessile drop) when respective experimental uncertainties are taken into account (**Table II**) (21–36). The discrepancies observed in the temperature coefficients could be attributed to the difference in processing techniques and the extent to which evaporation losses have been considered. The containerless approach presented in this report operates under high vacuum, isolating the reactive molten samples from container walls and gases and can process samples in such a short time (<3 s for Pt and Rh; <2 s for Pd, Ru and Ir, and <1 s for Os) that evaporation is not an issue. This is supported by measurements of the sample mass before and after the experiment that lead to values within the uncertainties of the balance (0.1 mg). Evaporation rates were not measured in these experiments but could be obtained by levitating a sample for a long time (for example, 15 minutes) as explained elsewhere (37). The conventional methods often imply chemical reactions between the melt and a crucible, a support or residual gases. This can alter the final density values.

Other possible sources of error could arise from the imaging technique (for example, optical focusing or image digitisation) and the measurements of sample mass. Material purity, oxygen solubility in the samples and gasification could also, to some extent, explain the discrepancies. Temperature measurements too are subject to errors and are complicated by the fact that emissivity data are scarce for metals above their melting temperature and, to our knowledge, have not been reported for undercooled materials (38). The interested reader can find a detailed analysis for each of the pgms elsewhere (4–6, 8, 10, 11) together with a complete comparison with the literature data, when available, obtained with conventional methods (21–36).

3.2 Surface Tension

The surface tension was measured over large temperature ranges, well above the melting temperature and down into the undercooled region as shown in **Figure 5** for all pgms except Pd (4, 5, 8, 10, 11). Although the density as a function of temperature was known and the radius could be tracked in real time, severe evaporation for Pd samples hindered these measurements because of the time needed to perform the experiments (one hour) and because the window of the vacuum chamber as well as the electrodes of the levitator became coated which made the sample unstable.

The uncertainty of the measurements was estimated to be better than 5% from the response of the oscillation detector and from the uncertainty in density measurements. In all measurements, the surface tension exhibited a linear behaviour as a function of temperature. The data, including those reported in the literature, are listed in **Table III**. At the melting point, the values obtained by electrostatic levitation (4, 5, 8, 10–12) show very good agreement with most published values when the respective experimental uncertainties are considered (22, 28, 32, 34, 36, 39–41) and the
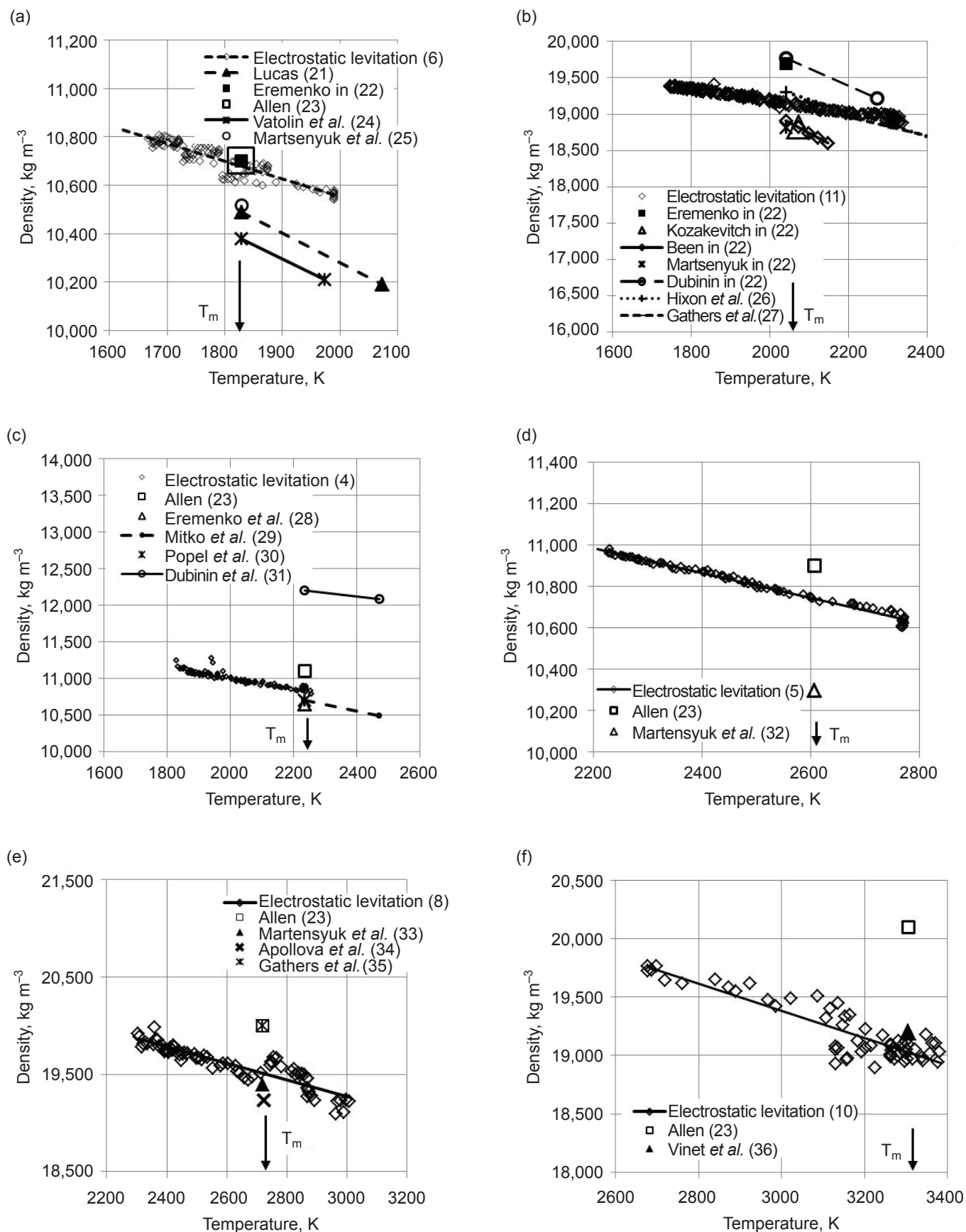
Fig. 4. Representative density data of equilibrium and non-equilibrium liquid pgms measured with an electrostatic levitator and compared with literature values as a function of temperature: (a) Pd (6, 21–25); (b) Pt (11, 22, 26, 27); (c) Rh (4, 23, 28–31); (d) Ru (5, 23, 32); (e) Ir (8, 23, 33–35); and (f) Os (10, 23, 36)

Table II Summary of the Density Data for Platinum Group Metals Measured by Electrostatic Levitation (4–8, 10, 11) Compared with Literature Values (21–36)

| Metal	T_m, K	Density, ρ		Temperature range, K	Reference
		ρ(T_m), 10^3 kg m^{-3}	dρ/dT, kg m^{-3} K^{-1})		
Pd	1828	10.66	–0.77	1640–1875	Electrostatic levitation (6)
		10.49	–1.226	1828–2073	Lucas (21)
		10.70		1828	Eremenko in (22)
		10.70		1828	Allen (23)
		10.379	–1.169	1828–1973	Vatolin *et al.* (24)
		10.52		1828	Martsenyuk *et al.* (25)
Pt	2041	19.2	–0.96	1691–2216	Electrostatic levitation (11)
		19.7		2041	Eremenko in (22)
		18.82		2073	Kozakevitch in (22)
		18.91	–2.882	2041–2148	Been in (22)
		18.81			Martsenyuk in (22)
		19.77	–2.4	2041–2473	Dubinin in (22)
		19.3	–1.7	2041–5100	Hixson *et al.* (26)
		19.1	–1.3	2095–4500	Gathers *et al.* (27)
Rh	2237	10.82	–0.76	1820–2250	Electrostatic levitation (4)
		11.1		2236	Allen (23)
		10.65		2236	Eremenko *et al.* (28)
		10.7	–0.90	2236–2473	Mitko *et al.* (29)
		10.7		2236	Popel *et al.* (30)
		12.2	–0.50	2236–2473	Dubinin *et al.* (31)
Ru	2607	10.75	–0.56	2225–2775	Electrostatic levitation (5)
		10.9		2607	Allen (23)
		10.3		2607	Martsenyuk *et al.* (32)
Ir	2719	19.87	–0.71	2300–3000	Electrostatic levitation (7)
		19.5	–0.85	2300–3000	Electrostatic levitation (8)
		20.0		2719	Allen (23)
		19.39		2719	Martsenyuk *et al.* (33)
		19.23		2723	Apollova *et al.* (34)
		20.0		2719	Gathers *et al.* (35)
Os	3306	19.10	–1.16	2670–3380	Electrostatic levitation (10)
		20.10		3306	Allen (23)
		19.2		3306	Vinet *et al.* (36)
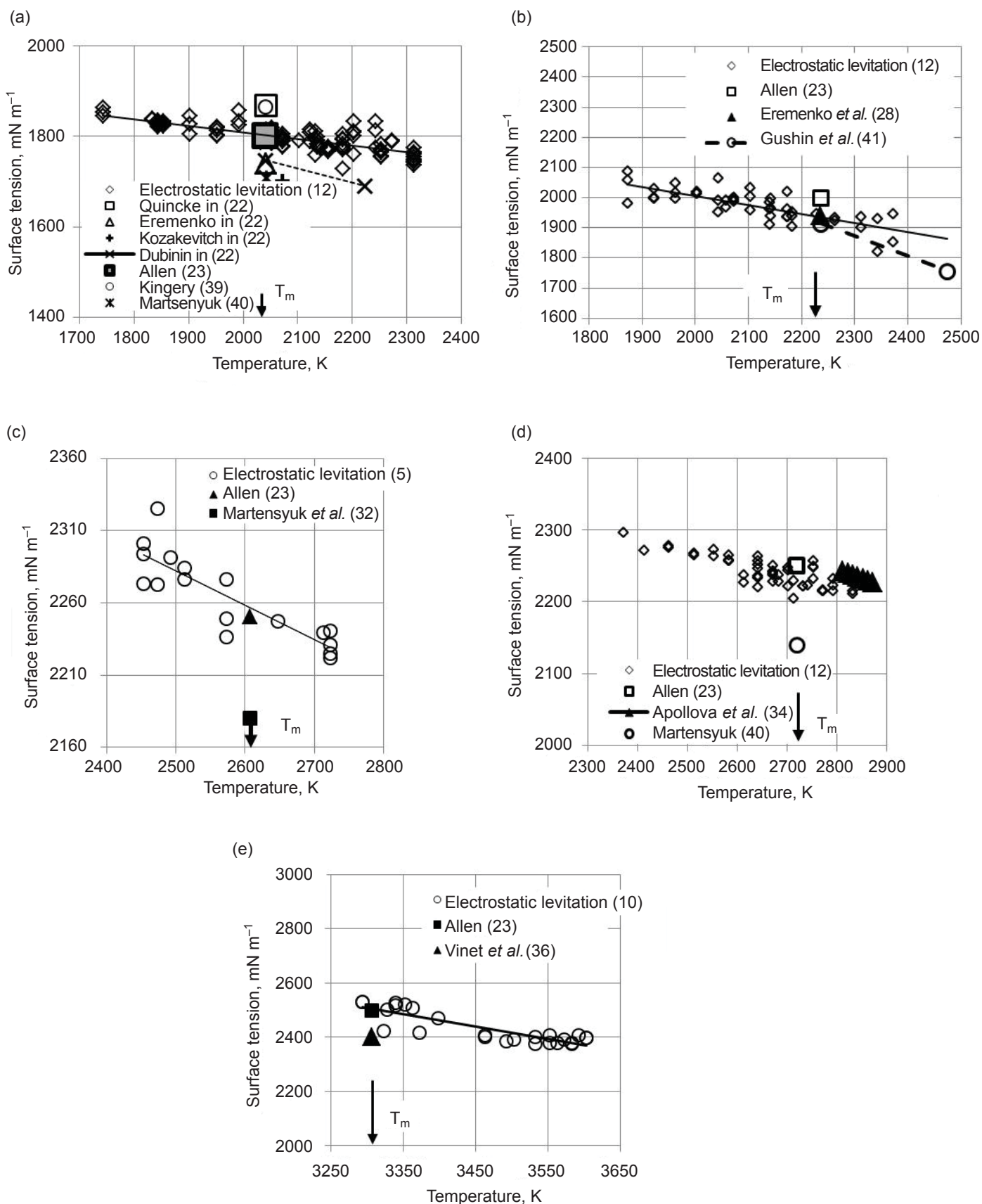
Fig. 5. Representative surface tension data of equilibrium and non-equilibrium liquid pgms measured with an electrostatic levitator and compared with literature values as a function of temperature: (a) Pt (11, 22, 23, 39, 40); (b) Rh (4, 23, 28, 41); (c) Ru (5, 23, 32); (d) Ir (12, 23, 34, 40); (e) Os (10, 23, 36)

Metal	T_m, K	Surface Tension, γ		Temperature range, K	Reference
		$\gamma(T_m)$, 10^{-3} N m^{-1}	$d\gamma/dT$, 10^{-3} N m^{-1} K^{-1}		
Pt	2041	1800	−0.14	1743–2313	Electrostatic levitation (11)
		1799	−0.17	1771–2141	Electrostatic levitation (12)
		1869		2041	Quincke in (22)
		1673		2053	Quincke in (22)
		1740		2041	Eremenko in (22)
		1699		2073	Kozakevitch in (22)
		1800		2041	Allen (23)
		1746	−0.307	2041–2148	Dubinin in (22)
		1865		2041	Kingery (39)
		1707		2043	Martsenyuk (40)
Rh	2237	1940	−0.30	1860–2380	Electrostatic levitation (4)
		1925	−0.28	2041–2403	Electrostatic levitation (12)
		2000		2237	Allen (23)
		1940		2237	Eremenko (28)
		1915	−0.664	2237–2473	Gushchin *et al.* (41)
Ru	2607	2256	−0.24	2450–2725	Electrostatic levitation (5)
		2269	−0.27	2275–2773	Electrostatic levitation (12)
		2250		2607	Allen (23)
		2180		2607	Martsenyuk *et al.* (32)
Ir	2719	2241	−0.16	2373–2833	Electrostatic levitation (7)
		2262	−0.28	2414–2808	Electrostatic levitation (12)
		2250		2720	Allen (23)
		2264	−0.247	2720–2873	Apollova *et al.* (34)
		2140		2720	Martsenyuk (40)
Os	3306	2480	−0.34	3230–3605	Electrostatic levitation (10)
		2500		3306	Allen (23)
		2400		3306	Vinet *et al.* (36)

Table III Summary of the Platinum Group Metals Surface Tension Data Measured by Electrostatic Levitation (4, 5, 8, 10–12) Compared with the Literature Values (22, 23, 28, 32, 34, 36, 39–41)
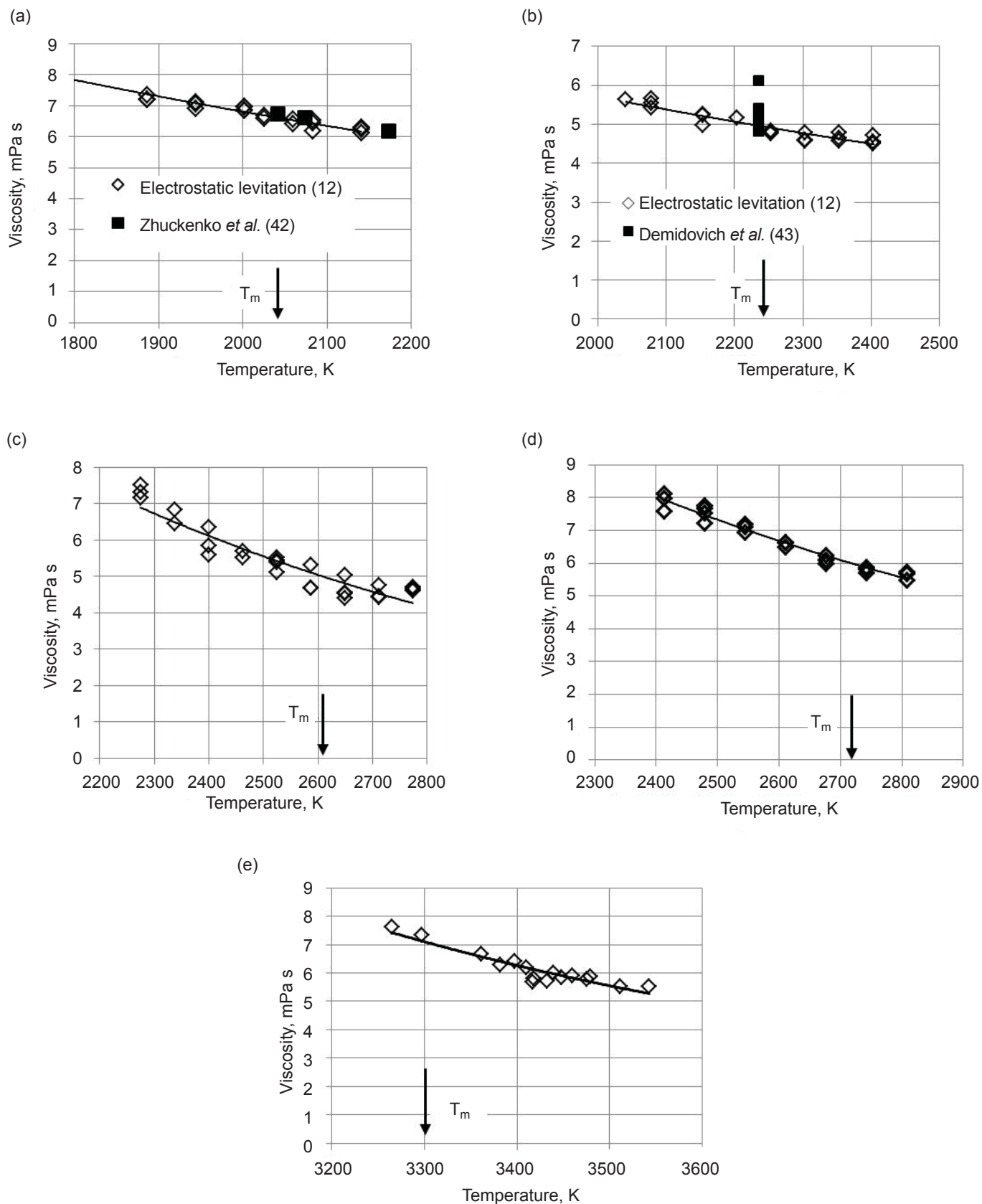
Fig. 6. Representative viscosity data of equilibrium and non-equilibrium liquid pgms measured with an electrostatic levitator and compared with literature values as a function of temperature: (a) Pt (11, 12, 42); (b) Rh (4, 12, 43); (c) Ru (5, 12); (d) Ir (8, 12); (e) Os (10, 13).

Table IV Summary of the Platinum Group Metals Viscosity Data Measured by Electrostatic Levitation (4, 5, 8, 10–13) Compared with the Literature Values (42, 43)

Metal	T_m, K	Viscosity, η			Temperature range, K	Reference	Note
		$\eta(T_m)$, 10^{-3} Pa s	$\eta = \eta_0 \exp(E/RT)$				
			η_0, 10^{-3} Pa s	E, 10^3 J mol^{-1}			
Pt	2041	4.82	0.25	49.9	1743–2313	Electrostatic levitation (11)	
		7.1	1.72	22.8	1771–2141	Electrostatic levitation (12)	(a)
		6.74	1.53	25.263	2041–2273	Zhuchenko *et al.* (42)	
Rh	2237	2.9	0.09	64.3	1860–2380	Electrostatic levitation (4)	
		4.9	1.31	24.7	2041–2403	Electrostatic levitation (12)	(a)
		5			2236	Demidovich *et al.* (43)	
Ru	2607	6.1	0.60	49.8	2450–2725	Electrostatic levitation (5)	
		5.0	0.47	51.2	2275–2773	Electrostatic levitation (12)	(a)
Ir	2719	7.0	1.85	30.0	2373–2773	Electrostatic levitation (8)	
		6.0	0.59	52.2	2414–2808	Electrostatic levitation (12)	(a)
Os	3306	4.2	0.0017	220	3230–3605	Electrostatic levitation (10)	
		7.0	0.098	117.5	3265–3542	Electrostatic levitation (13)	(a)

(a) Denotes the improved measurement procedure (20)

temperature coefficients compare generally well with those calculated by Allen (23).

Here again, the discrepancy observed between the results (values at the melting temperature as well as temperature coefficients) obtained with electrostatic levitation and other methods could be understood due to the fact that the containerless approach in high vacuum isolated the samples from container walls and gases, whereas the other methods imply possible chemical reactions between the reactive melts and a crucible, a support or residual gases. Melt contamination is possible and can drastically impact the surface tension. Sources of discrepancy or contamination include material purity, oxygen solubility in the samples, gasification and surface oxidation or nitridation from residual gases. Temperature measurements can also affect the end results. A full comparison with the data reported in the literature,

when available, as well as a complete analysis could be found in prior papers (4, 5, 8, 10–12).

3.3 Viscosity

The viscosity could also be measured over large temperature ranges, well above the melting temperature and down into the undercooled region as shown in **Figure 6** for all pgms except Pd (4, 5, 8, 10–13). Although the density as a function of temperature was known and the radius could be tracked in real time, severe evaporation for Pd samples again hindered these measurements because of the time needed to perform the experiments (one hour) and because the window of the vacuum chamber as well as the electrodes of the levitator became coated and made the sample unstable. The data shown in **Figure 6** were taken using a new procedure that consisted of measuring the viscosity on small samples and using a

lower feedback control frequency to minimise the effect on the damping oscillation (20). Data from the literature (Zhuchenko *et al*. (42) and Demidovich *et al*. (43)) are also superimposed in **Figure 6**. The temperature dependence of the viscosity exhibited an Arrhenius behaviour for all measured metals. The uncertainty of the measurements was estimated to be better than 15% from the response of the oscillation detector. The values of the levitation measurements (4, 5, 8, 10–13) and the very scarce literature data (42, 43) are summarised in **Table IV** for completeness.

4. Conclusions

The density, the surface tension and the viscosity of pgms in their equilibrium and non-equilibrium liquid phases were measured using the unique capabilities of an electrostatic levitation facility. The data obtained with electrostatic levitation were compared with those reported in the literature. The density and surface tension data summarised in this report highlight that measurements were taken over wide temperature ranges and that electrostatic levitation alone can offer values in the undercooled region. Furthermore, for refractory pgms (Ru, Ir, Os), electrostatic levitation is the only technique that can provide density and surface tension data over a temperature span covering even the undercooled region. Electrostatic levitation is also the only technique that can generate viscosity data for Ru, Ir and Os. The method not only gives a value at the melting point, but offers data for the superheated and the undercooled phases. Ongoing efforts focus on measurements of surface tension and viscosity of Pd to complete the series of property measurements of the pgms.

Acknowledgments

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