Numerical method of thermal shock resistance estimation by quenching of samples in water

A. G. LANIN, A. L. TKACHEV

Scientific Institute of SIA "LUCH", Podolsk, Moscow Region, 142100, Russia E-mail: iifedik@npoluch.msk.su

A temperature dependence of a transient heat transfer for cylindrical and ball samples (of different surface roughness) of 3–60 mm diameters heated up to the temperature range from 150 to 1200◦ C and quenched in a water bath of large volume was established. The measurement errors of the transient heat transfer defined by different methods with regard to hysteresis and statistical nature of boiling phenomena were evaluated. The study revealed, that the transition point from bubble to film boiling and vice versa differs essentially. The transient heat transfer in the field of bubble boiling did not depend on the size and the shape of the samples, their surface roughness and thermo-physical properties. But the magnitude of hysteresis in changing between the boiling regimes were substantially governed by the geometrical and thermo-physical characteristics of the samples. The examples of thermal stresses estimation which caused quenching damage to ZrC samples, heated up to a wide range of temperature from 150 to 1200 C, are given. The obtained data on the transient heat transfer and proposed recommendation on the temperature regimes of quenching for convenient sample sizes can form a basis of a standard for the numerical evaluation of the thermal shock resistance. © 2000 Kluwer Academic Publishers

1. Introduction

The thermal shock test method using a water bath is well established [1] and has attracted the attention of many scientists $[2-10]$ due to its experimental simplicity. Nevertheless, any quantifying estimate of thermal stresses which cause the testing specimens to fail is rather complicated because of intricate dependencies of heat transfer during water boiling on the surface of quenched samples.

The thermal shock resistance (TSR) is estimated [1–4] on the evidence of the maximum temperature difference ΔT between the surface temperature of the sample T_s and the water temperature T_w with regard to the heat transfer according to the Biot number $Bi = hr/\lambda$; taking the Bi to be constant during the quench test (where *h* is the heat transfer coefficient, *r*-characteristic size of specimen, λ-thermal conductivity). In practice, the heat transfer coefficient is an intricate function of the surface temperature of specimen and the assumption of constant *h* is responsible for the discrepancy of about 20% in the calculation of the temperature difference $\Delta T_s = T_c - T_s$ between the centre (T_c) and the surface temperature (T_s) of the sample at the given value Bi = 1 [2]. The errors of the ΔT_s calculation depends on the chosen value of *h* to a greater extent than the errors on the assumption of *h* constancy.

The maximum stationary coefficient *h* and the critical heat flow q_c for the bubbling regime ($T_w = 100^\circ$ C) on the surface of a body at atmospheric pressure are respectively equal to 3 \times 10³ W/m²K and 1,5 \times 10⁶ W/m² [11]. The q_c increases to 6×10^6 W/m², when the water

is at 20 \degree C. The increase of $q > q_{c1}$ transforms the bubble heat transfer to the steady film heat transfer regime thus decreasing the value of *h* to 1.2×10^3 W/m²K [11]. It should be mentioned that the critical heat flow q_{c1} in going from bubble to film boiling is considerably greater than the critical heat flow q_{c2} corresponding to the back transition. Such a hysteresis of the critical flow leads to considerable change of heat transfer and this must be given proper weight in determination of the TSR.

Transient heat transfer has been determined on the basis of temperature data measured by a fine thermocouple fixed in a silver ball of the diameter $d = 20$ mm quenched in water from different temperatures. The maximum transient value $h = 2.8 \times 10^4$ W/m²K was achieved at 250° C [12]. Further increase of the temperature to above 500 \degree C lowered *h* sharply to 2×10^3 $W/m²K$, that is typical for the film boiling. The measurement of temporal temperature changes of heated thin plates 1 and 3,2 mm in thickness made of zirconium gave the maximum value *h* in water of 1×10^5 W/m²K at 280–300 °C [9]. The measurement of transient heat transfer in silver and steel cylinders 6–15 mm in diameters with fixed into them thermo-couples gave the maximum values *h* in water in the range of $1,5-2,5 \times 10^5$ W/m²K depending on the sample diameters in the temperature field of $400-450$ °C [10].

The values of $h = 5.4 \times 10^4$ to 10⁵ W/m²K are of frequently used in the thermal shock testing with little justification [4]. The value of $h = 5.4 \times 10^3$ $W/m²K$ (which is more likely characteristic of natural convection) is taken in [7] without specifying the temperature interval of specimen heating in quench tests. The maximum values of *h*, calculated on the basis of experimentally obtained data, for the temperature difference ΔT , which caused sample failure, lay in the range $1-4 \times 10^{4}$ W/m²K [5, 8]. The authors [4, 5, 8] asserted that the thermo-physical properties, shape and size of samples, their surface condition, influence on the heat transfer and, as a consequence, the currently available dependencies of $h = f(T)$ are inadequate for reliable estimates of the TSR.

The goal of the paper is to establish the temperature dependencies of the heat transfer for the transient cooling in water of samples quenched from a wide temperature range. Furthermore, consideration is given to a possible heat transfer hysteresis and to an estimation of the reliability of the quantitative thermal shock quench tests.

2. Methods of the nonsteady heat transfer measurement

The determination of the nonsteady heat transfer on the surface of hot specimens quenched in a large volume water was performed by:

a) The measurement of the temperature in the centre of a small specimen during cooling when $Bi < 0.4$.

b) The measurement of the temperature fields registered for a period of time by a system of thermo-couples fixed in a large specimen.

c) The measurement of temperature over a period of time by one or two thermo-couples fixed in small or large specimens of known thermal-physical properties. The heat transfer in this case is determined by solving the inverse problem of the thermal conductivity [13].

The specimens as balls, solid and hollow cylinders having the diameter from 2 to 60 mm made of Cu, Ni, stainless steel, graphite with different surface finish, were used to measure the heat transfer. The temperature was measured in all tests by Chromel-Alumel thermo-couples $(d = 0, 1 \text{ mm})$ covered with an insulating layer. The thermo-couples were soldered using a silver alloy to the inner wall (0,5 mm in thickness) of the hollow cylinder. The specimens as hollow cylinders were thermally insulated and tightly sealed using a fitment (Fig. 1a) in order to prevent the heat transfer from the tube face surface. The junction of the thermocouples was fixed in a drilled orifice of the solid cylinder filled with an In-Ga liquid alloy (Fig. 1b). The insulated thermo-couples were incorporated into the graphite ball sample $<$ 60 mm in diameter using a mixture of furfuryl alcohol and graphite powder followed by curing and graphitization at a high temperature. The thermo-couples junctions were pinpointed from X-ray photographs made in three projections. The schematic of the setup to measure the thermal heat transfer, is shown on Fig. 2. A sample (1) is heated in an inert atmosphere of a furnace (2), the temperature, measured by the thermo-couple (7) was recorded by a logging potentiometer (8). A device (11) to automatically transfer the sample into a water-bath (3) and an oscilloscope (6) for thermometering the sample during quenching were switched on simultaneously with the help of a relay (9), when a preset temperature was attained. The shift of the boiling regimes was fixed by the change of the amplitude of the acoustic emission signal detected by a transducer (12) operating in the frequency range 1–20 of Kc.

The so called exponential method [14] derived from the time varying measurements of the sample temperature at small values of Bi $(0.4) was used as a basic$ method. In this special case, the temperature in the sample's centre somewhat differed from the surface temperature and the quantity of heat *Q* from the cooling surface *S* of a body having an *M* mass and a *C* heat capacity in a time $d\tau$ is expressed by the relation:

$$
Q d\tau = MC dT = h(\tau)(T_s - T_w)S d_\tau \qquad (1)
$$

and the coefficient of heat transfer is given by:

$$
h(\tau) = \left(\frac{MC}{S}\right) \frac{dT}{d\tau} \left[\frac{1}{(T_s - T_w)}\right]
$$
 (2)

where T_s and T_w are respectively temperatures of the sample's surface and water.

Figure 1 The fitments for the measurements of the heat transfer on the ring (A) and cylindrical (B) samples. 1. Ring sample 2. Asbestos sealings 3,4. Lower and upper covering 5,8. Thermo-couple with electroinsulation 6. Clamp 7. Cylindrical sample 9. Plug 10. Alloy Indium-Gallium.

Figure 2 The schematic of the set-up for the measurement of the heat transfer. 1. Sample 2. Furnace 3. Water bath 4, 10. Amplifier 5. Detector 6. Oscillograph 7. Thermo-couple 8. Potentiometer 9. Relay 11. Device for sample delivering 12. Transducer.

The typical curves of the temperature change in the hot sample quenched in the water-bath are presented in Fig. 3a and b. The smooth temperature curve in Fig. 3b attests the existence of pure bubbling on the surface. The drastic point of inflection *F* on the temperature curve (Fig. 3a) and the increase in the amplitude of the acoustic signals are evidence of the regime change from film to bubble boiling. The error ε of the heat transfer determination as a consequence of using the temperature in the centre of the sample T_c instead of the mean temperature T_m through the whole sample volume in the heat flow calculation $q = Q/S$ is:

$$
\varepsilon = T_{\rm m} - T_c'/T_{\rm m}' \tag{3}
$$

where $T'_{\rm m}$ and $T'_{\rm c}$ are temperature derivatives. The error of the heat transfer measurement did not exceed 10% for the copper cylinder with $d = 3$ mm, when Bi < 0,2 after the initial period of cooling longer than 0,01 s, as evident from the solution of the thermal conductivity equation [14]. The accumulated root-mean square error $\varepsilon_{\rm g}$ due to graphical differentiation of the temperature curves (5%), errors of temperature (2%) and of thermophysical properties (6%) measurements did not exceed 15% at a 64% confidence level.

When the second of the above mentioned methods for the heat transfer measurement is used, the temperature field throughout the graphite ball $(d = 60$ mm) is reconstructed using temperature data from 5 thermo-couples fixed in the sample. The surface temperature T_s can be deduced from the readings of two thermo-couples near the surface by two means: graphical extrapolation and calculation based of the assumption that the temperature between two extremes is governed by the cube parabola. The discrepancy in the determination of T_s by the two means do not exceed 2% and the accumulated measurement error *h* compared favourably with the error of the exponential method. The heat rate method through solving the inverse problem of the thermal conductivity [13] was used to determine the influence of the sample size, shape and properties on the heat trans-

Figure 3 The cooling of the heated copper cylinder with the diameter 3 mm in water bath ($T_w = 25^\circ$ C) with transition from film to bubble boiling (a) and without it (b).

fer and temperature change between the film and the bubble boiling.

3. Results and discussion

3.1. The measurement of transient heat transfer

The determination of heat transfer has been performed using a statistically representative measurement body because of a complicated character of a two-phase heat transfer connected with numerous hydraulic and thermodynamic factors. The measurement data are represented partially in Tables I and II. The transient heat transfer in quenching hot samples from the temperature level $T_0 = 250$ °C occured exclusively through bubbling, as in Fig. 3b. When the samples were heated in the range 250–350◦ C the heat transfer may occur both through pure bubble boiling and also through alternating boiling regimes (point F, Fig. 3a). The heat transfer

TABLE I The values of heat transfer on the mixed h_1 and bubble boiling h_2 regimes when the copper cylinder with $d = 3$ mm ($T_{cr} = 400$ °C at $T_0 = 500\textdegree C$) is cooled down from the temperature T_0 in the water bath ($T_w = 25\textdegree C$)

\boldsymbol{N}	$T \circ C$	$T_1 \circ C$	$q_1 \times 10^{-6}$ W/m ²	$\tau_1 \times 10^2$ s.	$h_1 \times 10^{-4}$ W/m^2K	$(T_1 - T_2)/T_m$ °C	$q_2 \times 10^{-6}$ W/m ²	$h_2 \times 10^{-4}$ $W/m^2 K$
17	200	200				$(200 \div 170)/190$	6,3	3,0
10	250	250				$(250 \div 170)/230$	7,9	3,4
19	250	242	$1,2 \pm 0,15$	1,3	0.5	$(242 \div 160)/220$	8,0	3,4
14	300	300				$(300 \div 280)/290$	10,5	3,75
18	300	285	1.4 ± 0.2	1,7	0.5	$(281 \div 230)/270$	10,5	3,75
13	350	350				$(350 \div 300)/325$	11,3	3,5
14	350	320	$1,6 \pm 1,1$	1,9	0.5	$(320 \div 280)/310$	10,8	3,5
19	400	380	1.7 ± 0.5	2,6	0,44	$(360 \div 320)/340$	10,7	3,1
16	450	400	1.9 ± 0.2	2,9	0,44	$(380 \div 350)/360$	10,7	3,0
10	500	400	2.0 ± 0.1	3,5	0,43	$(370 \div 350)/360$	10,7	3,0

N is the number of test samples, T_1 is the completion temperature of mixed boiling regime, q_1 is the heat flow and τ_1 is the medium life time of mixed boiling, T_m median temperature for the assignment of q_2 and h_2 in temperature range T_1 T_2 .

TABLE II The thermal flow change q_2 (Mw/m²) against the temperature of water bath, *T*^w

		The location of the sample beneath the water level [mm]			
		130	100	80	
T_0 °C	T_w °C	Thermal flow, $MW/m2$			
200	20	3.9	3,9	3,9	
200	100	0,8	0,8	1,0	
250	20	5,8	6,2	5,2	
250	50	3,5	3,1	2,9	
250	100	1,1	1,1	1,1	
300	20	8,0	7,7	7,2	
300	60	2,7	1,9	1,7	
300	80	1,5	1,5	1,5	
300	100	0,7	0.6	0,5	

occured exclusively on the schematic (Fig. 3a), when the sample temperature was above 350◦ C. The scattering of the peak heat flows under the mixed regimes dq_1/q_1 and the bubble boiling regime dq_2/q_2 and the lifetime of the mixed regimes $d\tau_1/\tau_1$ vary between 5 and 20% (Table I). The calculated values of h_1 and h_2 vary in the same ranges. The possibility of the bubble boiling with the heatup rate as high as 10^6 K/s at the temperature level above 350° C existed within a short period of cooling not longer than 30×10^{-6} s before the film formation [15].

Influence of water temperature and sample location in the water bath of large volume on the heat transfer rate was verified on a set of 5 copper rings (15 mm diameter, 10 mm height, 2 mm wall thickness), hermetically sealed and thermal-insulated as in schematic (Fig. 1a). It was shown that the intensity of heat transfer at $T_0 = 300$ °C was reduced from 3.7×10^{-4} W/m²K to 0.5×10^{-4} W/m²K with the waterbath temperature rise from 20 to 100◦ C, and the *h* values were equal independently of the sample location (Table II) in the limits of inevitable statistical dispersion. The sample movement velocity change from 2 to 25 cm/s in the temperature range of the developed bubble boiling had no effect on the coefficient of heat transfer.

The coefficient of film heat transfer statistically varied between 0,3 and 0.7×10 W/m²K independently of the sample size and thermal-physical properties in accordance with [11, 12]. Transition from the film to the bubble boiling regime was detected by the critical temperature T_{cr} depending on the complex $rC_{\rm m}/\lambda_{\rm m}$ and initial temperature T_0 , where r is the characteristic size; C_m , λ_m are the average values of the heat capacity and thermal conductivity in the temperature range from T_0 to 100[°] C. T_{cr} lowered and the life time of film boiling was extended with decrease of the complex rC_m/λ_m and with rise of the initial temperature of the sample *T*0. The dispersion of these parameters lay within 20%.

An appropriate temperature dependence of the heat transfer (Fig. 4), based on experimental data of the three measurement methods should be selected on the basis of the T_{cr} value. A distinguishing characteristic of the obtained *h* values fits in the left arm of the heat transfer curve for the samples having various values of rC_m/λ_m in the temperature range of bubble boiling. The variation of the heat transfer intensity in the transition from the film to the bubble boiling is characterised by the right arms of the curves (Fig. 4), the onset of which is defined by the T_{cr} temperature. It should be

Figure 4 The dependencies of the unsteady coefficient of the heat transfer *h* verses T_0 when the samples with various rC/λ are cooled in the water bath of large volume ($T_w = 25^\circ$ C). 1. $T_{cr} = 600^\circ \text{C}$, 2. $T_{cr} = 500^\circ \text{C}$, 3. $T_{cr} = 400 °C$ 4. $T_{cr} = 300 °C$ and 5. $T_{cr} = 200 °C$.

particularly emphasised that change from the film to the bubble boiling occured at a lower body temperature as compared with the back change. This circumstance leaded to that the *h* maximum value achievable e.g., for the sample with $rC_m/\lambda_m = 0.3 \times 10^{-2}$ m²s/kg in the course of quenching from $T_0 = 900^\circ$ C was found to be equal to 2×10^4 W/m²K that is two times lower than the *h* when $T_0 = 400^\circ$ C. The observed effect of hysteresis has been extensively investigated for a static heat transfer process [11], while the transient heat transfer regime differs greatly from the static one. The maximum q_2 value (Table I) is twice of the $q_{cr} = 6 \times 10^6$ $W/m²K$ [11] for the static heat transfer. The temperature dependence of heat transfer obtained by the exponential method in [12] agrees qualitatively with our dependence $h = f(T)$. However the evaluated *h* values are markedly lower since the measurements were conducted on the silver cylinder, 20 mm in diameter, at $Bi = 1,3$, and the error was more than in our investigation. Data obtained on thin plates turned out closer to our results and trustworthy [9]. The *h* values worked out in [5, 8] using the fracture time data, as a rule, were lower than the actual maximum value, since the sample failure may occur at a time, when the maximum heat transfer coefficient had been not attained yet during cooling from T_0 . Because of this the temperature curves $h = f(t)$ were shifted to a lower temperature region as compared with the curves of Fig. 4. In our view, the gained results of the transient heat transfer investigations with faithful appraisal of data reliability allow to settle existing disagreements in literature data and discard approximate estimates of the water quench tests.

3.2. Quantitative appraisal of water quench tests

Thermal shock quench tests were performed using a set-up similar to that used in the heat transfer measurements (Fig. 2). The only feature was the use of an acoustic registration system to fix the failure time, incorporating a unit to suppress the noise induced by boiling. However, to entirely get rid of the noise was not possible and the sample failure was detected only if the bubbling regime was not too developed. In this connection, the cracking detection was performed in conventional ways: using the method of penetrating dye, sample weakening under bending test. The damage to electrically conductive samples was estimated from the conductivity change that is more convenient in the thermal cycle tests. The temperature T_0 of initial sample failure was appraised by stepwise raising the temperature every other 10 degrees. The computations of thermal stresses were performed on the temperature T_0 and the failure time τ_f elapsed from the onset of cooling or using the T_0 value. At first the temperature field in the sample was calculated by numerical method with computer solving the equation of heat conductivity under non-linear heat transfer boundary conditions, set by our measurements. The TSR was defined from the thermal stress σ_t which caused failure or from the value $\Delta T = T_m - T_s$, where T_m was the mean integrated temperature over cross section of a sample, T_s was the

Figure 5 Relative error of $\Delta R/R$ versus Biot value at uncertainty Bio: 1–50%, 2–20%.

surface temperature. The known criterion for the TSR for the cylindrical sample $R = \sigma(1 - \mu)/\alpha E$ is, in fact, equal to ΔT and $R = \Delta T (1 - \mu)$ for disk sample. The main error of the appraisal ΔT depended on the Biot value, which, in turn, was dictated by the error of *h* and λ measurements. Taking the uncertainty of Biot to be 20%, the relative error of the experimental value $\Delta R/R$ would occur within 2–10% (Fig. 5) for the samples with $d = 6$ mm and $\lambda = 5 - 50$ W/m²K. This analysis was performed for $Bi = f(T)$. The error of ΔT calculation increased when Biot was adapted to be constant [17].

The calculated estimation of local thermal stresses σ_1 leaded to a value not more than $10^{-2} \times \alpha ET_0$, when the local heat flow q_1 equaled in the magnitude to the bubble size and opposite in sign to the heat flow *q* uniformly distributed over the whole sample. This value, obtained by Laplas approximation method for half-space [16] was negligible compared with the thermal stresses after exposure to the integral heat flow *q*. The validity of the calculated appraisal is supported by experimental results indicating that the local variation in the surface temperature is not more than 6◦ C with the existence of steam bubble less than 10^{-4} sec [18]. By this means, the estimation of the TSR does not come against the intractable problem in the course of the intense bubble boiling. The results of the thermal shock quench studies correlate with the data measured by other methods (Table IV) for the verification of the measurement reliability. The choice of the zironium carbide was dictated by some reason. First its σ , ΔT and complex $E\alpha$ are constant in the temperature range from 20 to $1500\,^{\circ}\text{C}$ as well as its corrosion resistance is stable in boiling water. Second, there are available reasonably extensive experimental data, obtained by other methods the errors of which are not greater than that of quench test. The

TABLE III The dependence of critical temperature T_{cr} change from film to bubble boiling vs the initial sample temperature T_0 with regard of thermal-physical complex *rc*/λ

Material, size	Graphite, $d = 60$ mm	Ni, $d = 40$ mm	Ni, $d=12$ mm	Steel, $d=2$ mm	Cu, $d=12$ mm	Cu, $d = 3$ mm
rc/λ 10^2 $(\mathrm{m}^2\mathrm{s/kg})$	44	15	5	2,4	0,9	0,3
$T_0, {}^{\circ}C$		T_{cr} , °C				
1100		190	185	200		
1000		390	280			
950				320	220	200
900		500	420		300	230
800		550	470	410	400	300
700	620	600	530	480	450	400
600	600	540	570	520	470	430
500	500	500	490		450	400
400	400	400	400		395	390

TABLE IV Thermal shock resistance of materials measured by various methods

. I quench test method, II induction method of heating, III method of heating in melted tin, IV method of electron beam heating, V method of radiation heating, *N* the number of test samples.

observed distinctions of ΔT within 15–25% measured by various methods are due to natural property dispersion unique to brittle materials. The ΔT values, relating to the first evidence of the sample damage under the quench test agree wholly satisfactorily with the data of other tests if we consider only the first sign of a sample damage [23]. The thermal stresses σ_{ts} slightly exceed the tensile strength and equal to 0,32–0,57 of the flexure strength (Table IV).

The reliability of the predetermined transient dependencies of the heat transfer and the temperature hystersis of the boiling regimes is reinforced by the damage regularities of ZrC samples heated up to different initial temperature levels T_0 from 150 to 1200◦ C. The outlasted change from the film to the bubble boiling, observed at $T_{cr} = 200\degree \text{C}$, when copper samples, 3 mm in diameter were heated above $1000\degree$ C is supported by the strength drop absence. The data spread and the mean strength value after quench test at $T_0 = 1100 - 1200$ [°] C compared favourably with the initial values since the thermal stresses σ_t were below σ_{ts} (Fig. 6). The possibility of quench strengthening with temperature rise up

Figure 6 Change of thermal stresses σ_t and relative strength drop σ/σ_b on quenching of ZrC cylinders 2 mm in diameter and length 45 mm in water bath ($T_w = 25^\circ \text{C}$) at various initial temperature T_0 .

to 1200◦ C is eliminated since stress relaxation and development of compressive stresses become feasible only in heating above $1500°$ C and subsequent mild cooling, e,g. by gas blowing around the ZrC sample [24]. The strength drop had its maximum upon heating to 400–600 \degree C, when the thermal stress σ_t reached its peak. A sharp fall of damage was also observed when Al_2O_3 cylindrical samples with $d = 1$, 2 mm were quenched from $T_0 = 1300 - 1400$ °C, as the maximum strength drop occured in the temperature region of 500–600◦ C. Unfortunately, it should be noted that the established facts about the heat transfer drop and the thermal stresses fall on samples heated up to above 800 ◦C did not take proper account in some quench research thus producing distorted estimation of the TSR.

4. Conclusion

The accomplished investigations of the film and the bubble boiling regimes on a body surface heated up from 150 to 1200 \degree C have permitted to reliably establish the temperature dependence of the heat transfer. It has been shown that the coefficient of the heat transfer does not depend practically on the body size, its thermal-physical characteristics and the surface conditions in heatup to 400◦ C. This allows to employ the obtained temperature dependencies of the heat transfer for samples of different materials and sizes. The error of the quench tests has been not worse than 15% for the samples as ball, solid and hollow cylinder with regard of its finite length [22]. The choice of the sample size is conditioned by a reasonable degree of approximation to true size of products with their peculiar distribution of surface and volume defects. It is necessary to test as, a minimum, 7 and 25 samples for the appraisal of the mean thermal shock value and its distribution.

It is necessary to allow for a temperature hysteresis intrinsic to the heat transfer change from bubble to film boiling depending on sizes and thermal-physical properties, when the samples are to be quenched from above 600° C. The results of the quench tests may significantly be distorted if this phenomenon is neglected. The proposed recommendations may form a basis for a standard to quantify the TSR by quenching method.

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