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As Published
http://dx.doi.org/10.1115/IMECE2008-67788

Publisher
ASME

Version
Final published version

Accessed
Sun Apr 24 14:10:40 EDT 2016

Citable Link
http://hdl.handle.net/1721.1/60916

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EXPERIMENTAL STUDY ON QUENCHING OF A SMALL METAL SPHERE IN NANOFLUIDS

H. Kim*, J. Buongiorno, L. W. Hu, T. McKrell, G. DeWitt
Massachusetts Institute of Technology
Cambridge, MA 02139, USA

ABSTRACT
The objective of this research is to systematically investigate the quenching characteristics of a hot sphere in nanofluids. The experiments are carried out with a small (9.5 mm) stainless steel sphere with initial temperatures near 1000 °C. Alumina nanofluids and deionized water are tested at low volume concentrations (less than 0.1 % by volume) and saturated conditions (100 °C). The results show that the quenching behavior in nanofluids is nearly identical to that in pure water. Moreover it is found that some nanoparticles accumulate on the sphere surface during the quenching process. Such accumulation of nanoparticles on the surface promotes the destabilization of the vapor film in subsequent quenching experiments, thus accelerating the return to nucleate boiling at higher temperature than that in the clean surface case.

NOMENCLATURE
T_{center} Temperature at the center of the sphere, °C
T_{MHF} Temperature at the minimum heat flux point, °C
T_{max,slope} Temperature at the maximum negative slope point, °C

INTRODUCTION
A number of recent investigations on boiling of nanofluids showed that such engineered fluids can effectively delay departure from nucleate boiling (DNB) with respect to pure fluids [1-9]. It was found that the DNB heat flux enhancement is closely related to nanoparticle deposition, which significantly roughens the heater surface by changing the initially smooth surface to one with peaks and valleys. Moreover the deposition of oxide nanoparticles like alumina and titania significantly enhances the affinity (or wettability) of the cooling liquid to the surface. These changes in the surface alter the boiling heat transfer characteristics, e.g., they increase the value of the critical heat flux.

Park et al. [10] performed quenching experiments of a high-temperature sphere in alumina nanofluids to investigate the effect of the nanoparticles on film boiling heat transfer. They observed an interesting phenomenon: after a quenching experiment of a sphere in nanofluids, the unwashed sphere quenched more rapidly through transition boiling, apparently bypassing the film boiling mode. This result suggested that nanoparticle deposition on the sphere surface prevents formation of a stable vapor film around the sphere, which consequently promotes a more rapid quenching. However, the main focus in that study was to explore the effect of nanoparticles on film boiling heat transfer, not quenching.

At MIT we are investigating the use of nanofluids in safety systems for Light Water Reactors [11], and quenching phenomena play an important role in ensuring the coolability of the nuclear fuel following loss of coolant accidents in such reactors. Specifically, because during an accident the fuel can be initially very hot (>700°C), its rewetting occurs slowly through the development of a quench front which advances upward in the reactor core. The speed of the quench front and thus the peak temperature reached during the reflood transient depend on a combination of factors including film boiling heat transfer, wettability of the fuel surface by the coolant, and localized axial conduction within the cladding near the quench front. The use of nanofluids could afford a significant increase of the quench speed for two reasons. First, boiling-induced deposition of nanoparticles on the surface greatly enhances surface wettability. Second, deposited nanoparticles of high-conductivity material (e.g., alumina) improve localized axial conduction in the cladding near the quench front. The enhanced wettability especially is expected to increase the minimum heat flux temperature (Leidenfrost point) for the cladding and promote its rapid cooling.

As a first step in the feasibility assessment of nanofluids for use in nuclear reactor accidents, the quenching characteristics of a small metallic sphere in nanofluids are studied. Nanofluids with 0.001, 0.01, and 0.1% volume concentrations of alumina nanoparticles are tested at saturated conditions (100 °C) under atmospheric pressure. In this paper, the transient cooling curves (temperature vs time) during the entire quenching process in distilled water and in nanofluids are investigated.

*Corresponding author: Email: hdkim@mit.edu
EXPERIMENT

Experimental Setup

Figure 1 shows the details of a test sample for the quenching experiment. The test sample consists of a metal sphere, a thermocouple to record the temperature at the center of the sphere, and a reinforcing precision tube to mechanically support them. The reinforcing tube is connected to a 9.5 mm-diameter connecting tube via a tube fitting.

A stainless steel sphere of 9.5-mm diameter is used as the quenching sample. The sphere is drilled to the center having a hole stepped from 0.9 mm to 0.5 mm in diameter, as shown in Fig. 1. A 0.5 mm-diameter K-type sheathed ungrounded thermocouple is inserted to the bottom of the hole by friction fitting. The installation of the thermocouple via friction fitting ensures a good thermal contact with the sphere, and thus minimizes its response time so that the rapidly changing temperature history of the sphere is acquired correctly.

A reinforcing tube of 0.6 mm ID and 0.89 mm OD is inserted between the 0.5 mm-diameter thermocouple and the 0.9 mm-diameter hole to mechanically support the test sample. A staking technique – hitting the edge with a sharp tool – is used to connect the tube and the sphere (See Fig. 1).

The reinforcing tube is a path of conduction heat loss during the experiments, which should be minimized. In the present experiment the ratio of the diameter of the reinforcing tube to that of the sphere diameter is very low (0.09), so conduction losses are negligible. In addition, the length of the tube is one of the important design parameters because vigorous boiling on the surface when quenching occurs causes vibration of the sphere, which may disturb formation of the stable vapor film around the sphere. Based on an analysis of mechanical vibration of the rod-sphere system, the proper length of the precision tube is determined to be 20 mm.

Figure 2 shows the schematic of the experimental setup for the quench test. It consists of the test sample, the radiant furnace, the air slide, the quench pool, and the data acquisition system.

A radiant furnace having a maximum temperature of 1500 °C is used to heat the test sample. A DC power supply (25V, 150A) is used to power the furnace. A B-type sheathed thermocouple is mounted inside the furnace to monitor the temperature.

A pneumatic air slide moves the test sample between the furnace and the quench pool with the stroke length of 200 mm. Pressurized air near 600 kPa is used to operate the slide. The average downward velocity of the sample is about 0.5 m/sec, which is measured from a visualization of the moving sphere using a high speed camera. Resultantly the time to convey the sample from the furnace to the pool is about 0.4 sec. A 4-way solenoid valve is adopted to change the direction of the slide.

The quench pool is 95 mm × 95 mm rectangular vessel having depth of 150 mm, which has an effectively infinite thermal capacity with respect to the sphere. It is made of Pyrex glass for visual observation of the quenching phenomena. The quench pool is placed on a hot plate with the maximum power of 800 W. The temperature of the quench pool is maintained with the feedback control of the hot plate and a Pt-100 ohm RTD sensor immersed in the quench pool. The side wall is insulated to minimize buoyant convection due to heat loss through the wall.

A HP agilent 34980A data acquisition system and a PC are used for gathering and storing temperature data from the thermocouple within the sphere. The temperature data is acquired at a rate of 10 Hz which is fast enough to monitor the temperature history of the quenched sphere.

Nanofluids

In the present study, water-based nanofluids with alumina nanoparticles are selected, as these particles are most widely used in the previous investigations of heat transfer in nanofluids. Nanofluids with the desired nanoparticle concentrations are prepared by diluting the concentrated nanoparticle solution purchased from Nyacol with distilled water. The vendor-specified particle size is 50 nm. The nanofluids with 0.001, 0.01, and 0.1% volumetric concentrations of the nanoparticles are tested at saturated temperature under atmospheric conditions.
RESULTS AND DISCUSSIONS

Quenching in distilled water

Figure 3 shows the typical boiling phenomena during the quenching of a fresh sphere in pure water with initial temperature of 1000 °C. Frame (a) shows the film boiling immediately after the sphere is immersed into the fluid. The stable vapor film is formed around the sphere. The sphere is optically deformed due to diffraction at the vapor-liquid interface of the vapor film. In frame (b), the film boiling near the minimum heat flux location is shown with the stable vapor film. At frame (c), the vapor film starts to be destabilized due to some contact of the liquid with the surface at the bottom of the sphere. Frame (d) shows the collapse of the vapor film. Some area is in contact with the liquid while some is still covered with the vapor film. In frame (e), the most vigorous boiling is shown on the sphere surface, where the critical heat flux occurs. Finally in frame (f), nucleate boiling takes place with the discrete bubbles nucleating on the sphere.

Figure 3 Boiling phenomena on the hot sphere quenched in saturated water at 100 °C: (a) film boiling just after immersion; (b) film boiling near the minimum heat flux point; (c) the destabilization of the stable vapor film; (d) transition boiling; (e) nucleate boiling near the critical heat flux location; (f) nucleate boiling

Figure 4 Temperature history at the center of the heated sphere in saturated water at 100 °C. Points (a) ~ (f) are corresponded to frames (a) ~ (f) in Figure 3.

The several transitions in the boiling mode change the heat flux on the sphere surface, resulting in the center temperature ($T_{\text{center}}$)-time history shown in Figure 4. Film boiling starts immediately after the sphere is immersed into the fluid. $T_{\text{center}}$ decreases gradually in the film boiling regime, as the heat transfer coefficient is small. Approximately at 230 °C, $T_{\text{center}}$ starts to rapidly decrease having a significant change in the slope of the curve. This change is caused by collapse of the stable vapor film around the sphere as shown in Figure 3(d). At that moment the boiling mode changes from film boiling to transition boiling, which has a higher heat transfer coefficient. Near $T_{\text{center}} = 180$ °C the curve has its maximum negative slope, which corresponds to the critical heat flux point and the start of the nucleate boiling mode. Finally $T_{\text{center}}$ asymptotically converges to the temperature of the surrounding liquid (100 °C) as the sphere experiences nucleate boiling heat transfer and lastly single-phase convective heat transfer.

Quenching in nanofluids

Figure 5 shows a comparison of the transient cooling curves for a fresh sphere quenched in water and in nanofluids with nanoparticle concentrations of 0.001, 0.01, and 0.1 vol.%.
The quenching behavior in nanofluids is nearly identical to that in pure water within the test range of the present study. It was shown previously that at low concentrations (<0.1 vol%), thermal physical properties of alumina nanofluid are almost identical to those of pure water [8]. Therefore, the results indicate that the nanoparticles present in the fluid do not affect the quenching behavior of a fresh sphere.

**Effect of nanoparticle deposition on quenching**

It is well known that nucleate boiling in nanofluids makes the dispersed nanoparticles deposit on the heater surface [5-9]. As the spheres in our experiments also experience nucleate boiling during the quenching process, nanoparticle deposition occurs on their surface, as shown in Figure 6.

In order to investigate the effect of nanoparticle deposition on the quenching behavior, we reheated the sphere previously quenched in nanofluid, and quenched it in pure water. In this case the quenching behavior of the sphere is considerably different from the behavior of a fresh sphere in pure water or in nanofluids, as shown in Figure 7. The film boiling heat transfer is similar to the other cases, but departure from film boiling occurs at higher temperature. As a result, the total quenching time is shorter. It is clear that the nanoparticle deposition on the surface promotes the departure from film boiling, thus accelerating heat removal from the sphere.

![Figure 6 SEM images of the surface of (a) a fresh (clean) sphere and (b) a sphere quenched in 0.01 vol.% alumina nanofluid](image)

![Figure 7 Effect of nanoparticle deposition on quenching of a heated sphere](image)

![Figure 8 Results of repeated quenching of a sphere in (a) pure water and in (b) 0.1 vol.%, (c) 0.01 vol.%, and (d) 0.001 vol.% alumina nanofluids](image)
Figure 8 shows the temperature histories when a fresh sphere is repeatedly quenched in water or a nanofluid without replacement of the sample. The small data scatter in the curve for pure water (Fig. 8a) shows the experimental repeatability. The cooling curve for the first run in a nanofluid falls within the scatter of the pure water data, as seen in Figure 5. However, in the second run the curves for the 0.1 and 0.01 vol.% nanofluid tests shifted considerably to the left, which is due to nanoparticle deposition on the sphere surface during the first run. Furthermore, the repeated runs in nanofluids appear to move the cooling curve further to the left because of the continued nanoparticle accumulation on the surface.

Visualization of the quenching process for a sphere with accumulated nanoparticles reveals that nucleation of vapor bubbles occurs intermittently even at temperatures that are typically associated to the stable film boiling regime (Figure 9). This suggests that nanoparticle accumulation on the sphere surface destabilizes the vapor film, thus accelerating return to transition boiling at a higher temperature, which is consistent with the trends shown in Figure 8(b).

Figure 10 shows the sphere center temperature at the minimum heat flux point \( T_{\text{MHF}} \) and at the maximum negative slope location \( T_{\text{max slope}} \), for the sphere repeatedly quenched in pure water and 0.01 vol.% nanofluids. Those points correspond to the minimum heat flux and critical heat flux locations on the boiling curve, respectively. The repeated quenching in nanofluids increases \( T_{\text{MHF}} \) from 230 °C to 450 °C while the runs in pure water do not. This result supports the fact that the accumulation of the nanoparticles on the surface promotes the destabilization of the vapor film so that the sphere with the nanoparticle deposits enters the transition boiling mode at higher temperature.

Park et al. [10] suggested that the roughness due to accumulated nanoparticles on the sphere surface destabilizes the very thin vapor film (which is typically <100 μm). Takata et al. [12] showed that the super-hydrophilicity of a surface with a TiO\(_2\)-coating increases the minimum heat flux temperature by 100 °C above that of a non-coated surface. In our experiments the accumulation of alumina nanoparticles forms peaks-and-valleys structures of the order of a few microns on the surface, and increases surface wettability as well [6, 7, 13]. Therefore, in light of Park et al.’s and Takata et al.’s observations, it is plausible to assume that both the enhancement of microstructure and enhancement of wettability are important factors in promoting the departure from film boiling. However more work is necessary to draw a definitive conclusion.

**CONCLUSIONS AND FUTURE WORKS**

An experimental facility was designed and constructed to investigate the quenching characteristics of nanofluids for a 9.5 mm-diameter stainless-steel sphere with an initial temperature of 1000 °C. Pure water and nanofluids with 0.001, 0.01, and 0.1% volume concentrations of alumina nanoparticles were tested at saturated conditions (100 °C) under atmospheric pressure. The following results were obtained:

- The initial quenching behavior in nanofluids was basically identical to that in pure water in the range of nanoparticle concentrations from 0.001 to 0.1 vol.%.
Some nanoparticles deposited on the sphere surface during the quenching process. Subsequent re-quenching tests showed that such nanoparticle deposition accelerated the return from film boiling to transition boiling, which occurred at higher temperatures than in the fresh surface case. Specifically, after 4 re-quenching experiments the minimum heat flux temperature increased by >200°C. It is assumed that the physical mechanism responsible for this acceleration of the quenching process is the destabilization of the vapor film in film boiling, due to the accumulation of the particles on the surface.

In the future work a detailed analysis of the boiling heat transfer characteristics during the quenching experiments will be performed using the inverse heat transfer method to evaluate the surface heat flux and temperature, which will enable acquisition of the full boiling curve for the nanofluids. In addition, the nanoparticle deposited-surface will be carefully characterized to elucidate the mechanism by which the nanoparticle deposition layer destabilizes the vapor film in film boiling, and consequently promotes the return to the transition boiling mode.

ACKNOWLEDGMENTS
This research was supported by AREVA and a generous gift from Mr. Doug Spreng. This work was supported by the Korea Research Foundation Grant funded by the Korean Government (MOEHRD) (KRF-2007-357-D00026). The authors are also grateful to Roberta Concilio Hansson of the Royal Institute of Technology (Stockholm) for providing valuable advice on the design of the experimental apparatus, and reviewing the paper.

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