International Journal of Heat and Mass Transfer 119 (2018) 446–459

Contents lists available at ScienceDirect

International Journal of Heat and Mass Transfer

journal homepage: www.elsevier.com/locate/ijhmt

Cryogenic quenching of rock using liquid nitrogen as a coolant: Investigation of surface effects

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ARTICLE INFO

Article history:
Received 15 June 2017
Received in revised form 22 November 2017
Accepted 24 November 2017

Keywords:
Liquid nitrogen
Quenching
Rock
Film boiling
Leidenfrost point

ABSTRACT

Cryogenic quenching experiments were conducted using sandstone rock samples and liquid nitrogen (LN_2) under ambient pressure. Four different rock surface conditions were prepared to investigate the surface effects on quenching, as compared to the baseline case where rock surface was polished by 1500 grit sandpaper. A copper sample was also made to reveal the unique property of rock surface with respect to quenching. It is found that a 130 °C increase of LFP was achieved by rock surface than copper, when both were polished by 1500 grit sandpaper. The scanning electron microscope (SEM) images showed that numerous pores and cavities with hierarchical dimensions were present on rock surface. These cavities contributed to the LFP enhancement by providing massive nucleation sites for bubble formation when intermittent solid–liquid contact occurs during film boiling thereby destabilizing the vapor film. Rock surface polished by 36 grit sandpaper was found to be superhydrophilic and rougher than the baseline case but produced no enhancement on cryogenic quenching. Quenching rates were remarkably accelerated on rock surfaces with sand layer coating and with orthogonally intersected grooves. The initial room temperature was not enough to maintain stable film boiling on these two surfaces and transitional film boiling dominated the boiling regime after quenching started. Further increase of LFP was achieved on rock surface covered by crude oil, which served as a low thermal conductivity coating. The present results demonstrated that the unique surface micro structure of rocks can significantly raise LFP temperature thus shortening film boiling duration. In addition, by appropriately modifying the rock surface condition, cryogenic quenching rates and LFP can be further enhanced.

1. Introduction

As a typical cryogenic fluid, liquid nitrogen (LN_2) has been widely applied in various industrial sectors including aerospace systems, cryosurgery systems as well as the cooling of superconductivity cables [1,2]. In oil and gas industry, researchers are exploring a novel technique called cryogenic fracturing, which utilizes the cryogenic temperature of LN_2 to drastically cool down reservoir rocks. The resulted temperature gradients near rock surfaces are expected to induce tensile stress and to facilitate the fracturing of rocks so that oil or gases can readily flow through these fractures into well bores [3,4]. Rocks will experience cryogenic quenching from its initial temperature down to cryogenic temperature when brought into contact with LN_2. Complicated heat and mass transfer processes involving boiling of LN_2 and transient heat conduction within rocks will occur in a conjugate manner during the cryogenic quenching. An accurate understanding of the associated boiling heat transfer and its potential affecting factors is crucial as higher heat transfer rates are always desirable to generate sharper temperature gradients and larger tensile stresses in rocks.

The evolution of heat flux during quenching can be described as an inverse route along the boiling curve, which plots surface heat flux against wall superheat [5]. With the wall temperature decreases, quenching system will sequentially undergo film, transition and nucleate boiling, followed by single phase convection. Film boiling is associated with a remarkably low heat transfer rate thus is expected to be shortened in order to achieve an overall quenching acceleration [6]. A large amount of studies, most of which use water based fluids as quenching liquids, have been conducted in the open literatures to find ways to accelerate quenching. Among those methods, modification of quenching liquids or solid surfaces is most commonly seen. Firstly, quenching with nanofluids has been extensively studied using different nanoparticle types, concentration, with different solid materials, geometric shapes and under different liquid subcoolings [6–14]. One consensus of these studies is that quenching speed can be significantly enhanced during repeated quenching in nanofluids, where the deposition of

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https://doi.org/10.1016/j.ijheatmasstransfer.2017.11.123
0017-9310/

nanoparticles on solid surface plays an important role. The deposited nanoparticles tend to alter surface characteristics such as wettability, roughness and porosity, which can influence the Leidenfrost point (LFP) [15]. Kim et al. [6] revealed that intermittent liquid–solid contacts can occur even at film boiling when solid surfaces are covered by nanoparticles and this would lead to violent liquid vaporization which destabilizes the vapor film [9]. Fan et al. [12] conducted quenching experiments using saturated graphene-based aqueous nanofluids and stainless steel spheres. They found that at relatively high concentration (0.05 wt%), the graphene-based nanofluids could enhance film boiling heat transfer.

Apart from the utilization of nanofluids, efforts were also made to manipulate the physical and chemical properties of the quenching surface. Fan et al. [16] managed to control the wettability of surface by coating nanostructured layers on metallic spheres. The quenching of superhydrophilic sphere with water was greatly accelerated by the violent rewetting observed at film boiling compared to the hydrophilic baseline case. Critical heat flux (CHF) was increased by 70% for the superhydrophilic surfaces. Kang et al. [17] and Fan et al. [18] obtained superhydrophilic surfaces through anodic oxidation and silica nanoparticle coating methods. Their quenching experiments with water showed that film boiling heat transfer coefficient (HTC), LFP, and CHF were all improved with superhydrophilic surfaces. Particularly, transitional film boiling sub-regime was observed in Fan et al. [18]'s study and this sub-regime was due to point-contact between solid and liquid. Superhydrophilic surfaces can facilitate the point-contact by their strong wettability with water. Lee's group [19–21] investigated surface effects of metallic rodlets on the quenching performance with water. Cylinder rodlets of different materials with groove-structured or oxidized surfaces were used in quenching experiments. They found that the groove structures fabricated on solid surfaces can essentially contribute to earlier liquid–solid contact, thus lead to higher LFP and film boiling HTC. The authors attributed this effect to smaller volume-to-area ratio of the groove structures [19,21]. Kozlov and Keßler [22] also conducted quenching experiments using metallic rodlets with groove structures. Their results on steel rodlets showed that large groove tips can pierce vapor film and destabilize film boiling. Consequently, the vapor film would be disrupted earlier and quenching was accelerated. Due to the difficulties associated with cryogenic experiments, there are very limited studies on pool boiling quenching using LN_2 as a coolant. Hu et al. [5] quenched aluminum rodlets into LN_2 pool and investigated the effects of nanoporous surface structures. They found that the heat flux at nucleate and transition boiling and the CHF were substantially improved with nanoporous surface compared to smooth surface. Visualization analysis indicated that the nanoporous surfaces provided more nucleation sites for bubble formation [5], which may serve to the enhancement of boiling. Tsoi and Pavlenko [23] coated a copper plate with low thermal conductive coatings and quenched this plate into LN_2. Their experiment showed that quenching rate could be increased by low thermal conductive coating. The largest quenching rate enhancement was 2.6 folds with coating layer of 90 μm. More rapid surface temperature decrease caused by the additional thermal resistance was proposed as an explanation for the quenching enhancement.

Most, if not all, of the previous quenching studies focused on metallic materials, partly because metals are primary components of thermal systems encountered in industries. To the authors' knowledge, there have been no studies on quenching of rocks, especially using LN_2. Several researchers investigated heat transfer between LN_2 and concrete, aiming to evaluate the evaporation rates of cryogenic liquids spilled on ground [24–27]. However, on one hand, these studies failed to provide insights into the boiling regimes of LN_2 and affecting factors. On the other hand, as an artificial material, concrete is substantially differed from natural rocks with respect to chemical components and physical properties. As an important geo-material, rocks are natural aggregates of different minerals among which pore spaces are present [28]. As compared to metals, rocks may feature coarse surfaces and low thermal conductivity. It has been confirmed that solid surfaces can have profound influences on quenching behavior, thus the porous and rough surfaces of rocks are very likely to affect quenching process. This paper therefore studies cryogenic quenching of rocks using LN_2 through laboratory experiments. The purposes are to reveal cryogenic quenching characteristics of rock surfaces as well as to investigate the effects of surface conditions. If the cryogenic quenching of rocks by LN_2 can be enhanced then the cryogenic fracturing technique will be more practical and effective.

In Section 2, description of experimental setup is presented. Section 3 presents experimental results and discussion. Summary and conclusions are given in Section 4.

2. Experimental

2.1. Apparatus and procedure

Fig. 1 shows the schematic and photo of experimental setup employed in this work. Different from the spheres or rodlets widely used in quenching studies, we used flat plates made of rocks for cryogenic quenching experiments under atmospheric pressure. This is due to the simplicity of processing rocks into circular plates than into spheres or rodlets. As shown by Westwater et al. [29] plates and spheres are equally reliable for quenching studies. The experimental apparatus mainly consists of a cylinder double-walled stainless steel (SS) container, which is connected to a vacuum pump. By evacuating the container heat loss from the ambient air to the quenching system can be suppressed. The inner diameter of the SS container shrank at the bottom so that a perpendicular stage structure was formed. Rock samples used in this work are circular plates with nominal thickness of 30 mm and diameter of 80 mm (see Fig. 2a). The geometric size of rock samples accords with the principles proposed by Westwater et al. [29] to perform standard quenching practice. Sandstone containing fine mineral particles and with good homogeneity was chosen in this work and Fig. 2b shows photograph of as-received rock sample. The rocks were placed at the bottom of SS container to serve as boiling surface (see Fig. 1c). There was a 5 mm gap between rock sample and SS container inner wall and this gap was filled with Teflon, as shown in Fig. 1a. As a result, radial heat loss could be minimized due to low thermal conductivity (~0.2 W/m/K) of Teflon and the vacuum so that one-dimensional heat conduction in rocks was established. Jones et al. [30] pointed out that any small gaps or cracks around boiling surface can bring undesirable nucleation sites which may affect experimental precision. In present apparatus the interfaces between rock, Teflon and container inner wall may induce the above negative effects. To address this issue different methods were tried and we finally used glue water to cover the gaps. Two benefits can be obtained here: the glue water has relatively low viscosity and can flow so that a smooth surface can be established covering any small gaps (see Fig. 1a). In addition, the glue water gets frozen after encountering LN_2 therefore the gaps can be robustly insulated and no LN_2 could leak out of the pool.

T-type thermocouples (TCs) 1 mm in diameter and 0.1 K in precision were used to measure temperature changes during quenching. The response time of used TCs was less than 0.2 s, according to the manufacturer. To mount TCs the rock samples were drilled with three holes 2 mm in diameter. It was determined impractical