Time-resolved laser-induced-incandescence (TR-LII) for sizing of iron nano particles

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Time-resolved laser-induced incandescence (TR-LII) as well as rapid particle sampling and transmission electron microscopy (TEM) were applied to the size measurement of chain-like iron nanoparticles. From the measured TR-LII signals both, the thermal energy accommodation coefficient α_T and the mean particle diameter were determined. The TR-LII-measured particle size is in an excellent agreement with the TEM-determined primary particle size.

Introduction

The objective of the present work is to apply the TR-LII method to the size measurement of iron nano particles. To eliminate the influence of unknown optical properties, the particle temperature after laser heat-up was directly measured by two-color pyrometry. The model used for signal evaluation is described in [1].

Experiments

Iron particles are synthesized in a hot-wall flow reactor, where gaseous iron penta-carbonyl ($Fe(CO)_5$) is used as a precursor diluted in argon and nitrogen, respectively. Within the reactor, the precursor decomposes and nucleation of iron particles takes place [2]. At the exit of the reaction tube, the gas-particle-flow enters an optically accessible measurement chamber. The particles are heated by a pulsed Nd:YAG laser at a wavelength of 1064 nm. The signal is detected perpendicular to the laser beam via a beam splitter and two narrow (10 nm FWHM) band-pass filters at 550 and 694 nm in front of two fast photomultipliers. For particle-size evaluation, the time-resolved signal detected at 550 nm was used, while the signal at 694 nm is used to determine the initial particle temperature by two-color pyrometry. A pneumatic sampling system [3] allows to extract particle probes for TEM analysis. The experiments presented here were performed at p = 500 mbar and T = 670 K with 3200 ppm iron penta-carbonyl. Under these conditions, the magnetic properties of the iron particles cause the formation of chainlike particles [2].

Results

A series of normalized particle emission signals during particle cooling obtained from iron particles in argon with various laser energy densities is shown in fig 1. Although the measurements were performed at identical particle structure and gas conditions, the cooling behavior is obviously different. For an evaluation of the signals in terms of particle size, at first the translational thermal accommodation coefficient α_T had to be determined. For this task, α_T was used as an additional fit parameter together with the mean particle diameter during signal evaluation [1]. In both dilution gases α_T = 0.13 was determined for the iron particles.

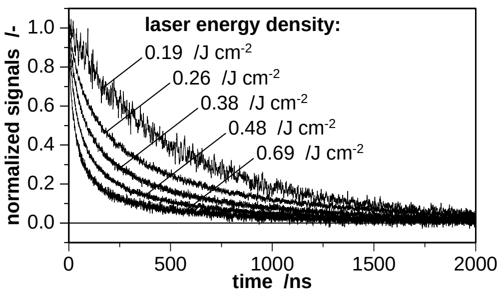

Fig. 1: Normalized TR-LII signals.

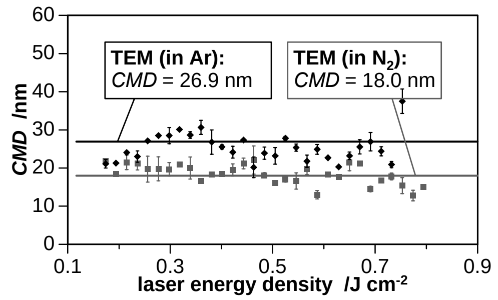

Fig. 2: TR-LII determined particle diameter for iron particles in argon (black) and in nitrogen (gray).

Fig. 2 shows the resulting mean particle diameters as a function of laser-energy density for iron particles in argon or nitrogen, respectively. The continuous lines give the results of the TEM analysis. The TR-LII determined particle size is obviously independent from the laser energy density and is in an excellent agreement with the TEM determined mean primary particle size.

References

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Proceedings of the International Bunsen Discussion Meeting 2005: Laser-induced Incandescence, Quantitative interpretation, modelling, application