



Lab Manual

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Contact Angle Measurement System

(kruss)

1.0 Title

Kruss contact angle measurement system

2.0 Purpose

The Kruss Contact Angle Measuring System is used to determine surface properties of solids and liquids using the sessile and pendant drop methods, respectively. The sessile drop method is an optical contact angle technique used to estimate wetting properties of a localized region on a solid surface. The angle between the baseline of the drop and the tangent at the drop boundary is measured. The pendant drop method is used to measure surface and interfacial tensions of liquids. Here, the geometry of a drop is analyzed optically.

Features of the Kruss contact angle measuring system include:

- ▶ Measurement of static and dynamic contact angles
- ▶ Zoom lens to optimize image size
- ▶ Integrated dosing system
- ▶ Integrated bright field illumination
- ▶ Sample stage can hold 4" wafers

Accessories include:

- ▶ Software DSA1 for fast and fully automatic contact angle and surface tension measurement of liquids and solids

3.0 Scope

This chapter describes the components of the contact angle measuring system, the operating software, and their usage.

4.0 Applicable Documents

[Revision History](#)

Krüss Drop Shape Analysis Manual (DSA)

5.0 Definitions and Process Terminology

6.0 Safety

Be careful when handling the syringe. Although it is not sharp, careless usage still presents a safety hazard. In addition, use caution when raising the stage, as to avoid hitting the end of the syringe and damaging the tip. Lower stage and raise the micrometer before removing the syringe to refill the syringe with D. I. water. This is to avoid damaging the tip of the syringe.

7.0 Statistical/Process Data

8.0 Available Process, Gases, Process Notes

9.0 Equipment Operation

9.1 Precautions and hints

The syringe contains deionized water. To use another liquid, contact the superuser for a syringe change.

For liquid-air and liquid-liquid interfacial tension measurements, and for contact angle measurements with test liquids other than water, see the superuser for assistance.

9.2 Start-up

9.2.1 Enable kruss on the wand.

9.2.2 Turn on the illumination, using the knob on the left side of the machine.

9.2.3 Double-click on the DSA program symbol on the Windows desktop to start the operating software.

9.3 Measuring contact angle of a liquid on a solid

9.3.1 In the sub-menu **Option | Drop Type** select the option **Sessile Drop**.

9.3.2 In the sub-menu **Option | Subtype** select the correct drop direction. For a drop which lies on a horizontal surface in the image, select **Normal Sessile Drop**.

9.3.3 Open the FG-Window to show the live image. Select **File | FG-Window**.

9.3.4 Press the video camera icon. The live image of the drop appears on the computer screen.

9.3.5 If the needle is not in the video image, the syringe holder can be adjusted by using the Allen wrench located on the desk. Make sure the needle can be seen in the video image, as the software uses the needle image as a reference.

9.3.6 Using the zoom control, select as large a magnification as possible while ensuring the tip of the needle remains on the screen. Sharply focus on the edge of the needle, and then zoom out to a medium magnification.

9.3.7 Produce a drop (~2 μL) by turning the micrometer slowly on top of the syringe. Make sure the micrometer is pressing against the top of the syringe. The drop should be approximately twice the diameter of the needle. Elevate the sample stage so that the drop touches the solid surface, and then lower the sample stage so the drop rests on the sample.

9.3.8 Adjust the illumination so that no reflections are visible on the drop image. The focusing assistant (click on the octagonal icon in the DSA software) can be used to check the sharpness of the image. The distance between the lens and the drop should be adjusted so that numbers in the focusing window are between 1 and 10.

9.3.9 Press the camera icon to snap the drop image, which should appear in the FG-Window.

9.3.10 Select **Option | Drop Info**. A menu window appears in which various test data, such as liquid densities and the diameter of the needle, can be entered. The default values are for water on a solid sample in air.

9.3.11 The lowest line in the FG-Window represents the sample surface. Move this baseline by using the mouse or arrow keys until it matches up with the sample surface. The left and right arrow keys can be used to change the tilt of the baseline. Also, for contact angles less than 90 degrees, the baseline can be determined automatically by activating the function Substrate Detection in the Option menu. (The upper two lines are used in measuring the magnification factor, which is needed in pendant drop measurements).

- 9.3.12 To determine the contact angle, select a method in the submenu **Profile | Contact Angle Using....** Four methods are available: **Tangent 1**, **Tangent 2**, **H/W-Method** (height-width method), and **Sessile Drop Fitting**. The H/W method gives reliable data for contact angles less than 90 degrees. The Sessile Drop Fitting method fits the drop shape to the Young-Laplace equation and is recommended for contact angles greater than 90 degrees. The contact angle can also be taken by pressing the icon that shows a drop on a surface with an angle measured.
- 9.3.13 The result appears in the result line at the lower margin of the screen. The result can be displayed with the densities and other test parameters by pressing the table icon. This opens the Result Window with the current measurements. For a plot of the results, press the plot icon, which opens the Plot Window with the current measurements.

9.4 Transfer Data Files (result)

Note: Make sure the power switch is pointing the white dot on the power strip and both 432B1 & kruss Dell computers are power on.

9.4.1 Save and transfer image file:

- 9.4.1.1 Click FG Window with drop image after determining the contact angle.
- 9.4.1.2 Click **File** on top menu bar and choose **Save As**.
- 9.4.1.3 Choose **My Documents** directory, find and open your own folder.
- 9.4.1.4 Files can be transferred from the kruss computer via floppy disk. Please remove files promptly to save space on the hard drive. All data files will be removed when disk space is low.

9.4.2 Save and transfer result measurements:

- 9.4.2.1 Click Result Window with the measurement files.
- 9.4.2.2 Click **Edit** and then **Select All** files on menu bar.
- 9.4.2.3 Click **Edit** and then **Copy** on menu bar.
- 9.4.2.4 Click **Start** button on the left bottom of the computer screen, select **Programs**, **Accessories** and click **WordPad**.
- 9.4.2.5 Click **Edit** on WordPad menu bar and then click **Paste**.
- 9.4.2.6 Click **File** and **Save As** on WordPad menu bar.
- 9.4.2.7 When desktop **My Documents** icon appears, find and open your own folder.
- 9.4.2.8 Create new folder name and save measurement file as a document (.doc) file.
- 9.4.2.9 Files can be transferred from the kruss computer via floppy disk. Please remove files promptly to save space on the hard drive. All data files will be removed when disk space is low.

9.5 Shutdown

Once measurements are complete, the shutdown procedure is as follows:

- 9.5.1 Exit DSA program and other window programs.
- 9.5.2 Turn off illumination.
- 9.5.3 Disable kruss on the wand.

10.0 Troubleshooting Guidelines

11.0 Figures & Schematics

12.0 Appendices

12.1 Non-Water Contact Angle Measurements

A wide variety of organic solvents (primarily alkanes and alcohols) are also used for characterization of surface wetting. You may use the Kruss to determine contact angle with liquids other than water. All non-water contact angle test fluids must first be reviewed with the Microlab Technology Manager ([Bill Flounders](#), bill at eecs dot Berkeley dot edu). The solvent present in the syringe is essentially sealed and the volume dispensed on the sample surface is de minimis – exceedingly small. Therefore, you are not required to have the tool in a fume hood when measuring contact angle with most organic solvents.

Syringe loading and any bulk solvent handling work must be performed in a fume hood. When finished with non-water test liquids, or between different solvents, rinse the syringe sequentially with isopropanol, then DI water, then dilute sulfuric acid, then again with DI water. Blow-dry with N₂. The Pyrex syringe and SS needle are compatible with all solvents typically used for contact angle measurements. Do not use any hydrofluoric acid in the system. You may purchase your own Hamilton syringes and use them with the system if you prefer.

12.2 Related References

Evaluation of Different Approaches to Assess the Surface Tension of Low Energy Solids by Means of Contact Angle Measurements, A. R. Balkenende, H.J.A.P. van de Boogaard, M. Scholten, N.P. Willard, Langmuir, Vol. 14, p. 5907-5912, 1998.

Contact Angles on Hydrophobic Solid Surfaces and Their Interpretation.

D. Li and A. W. Neumann, Journal of Colloid and Interface Science, Vol. 148, No.1, Jan. 1992.

Constitutive Relations in the Wetting of Low Energy Surfaces and the Theory of the Retraction Method of Preparing Monolayers. E.G. Shafrin, W.A. Zisman. Journal of Physical Chemistry, Vol. 64, No. 5, p. 519-524, 1960.