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The applications of statistical quantification techniques in nanomechanics and nanoelectronics

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Abstract
Although nanoscience and nanotechnology have been developing for approximately two decades and have achieved numerous breakthroughs, the experimental results from nanomaterials with a higher noise level and poorer repeatability than those from bulk materials still remain as a practical issue, and challenge many techniques of quantification of nanomaterials. This work proposes a physical–statistical modeling approach and a global fitting statistical method to use all the available discrete data or quasi-continuous curves to quantify a few targeted physical parameters, which can provide more accurate, efficient and reliable parameter estimates, and give reasonable physical explanations. In the resonance method for measuring the elastic modulus of ZnO nanowires (Zhou *et al* 2006 *Solid State Commun.* **139** 222–6), our statistical technique gives $E = 128.33$ GPa instead of the original $E = 108$ GPa, and unveils a negative bias adjustment f_0. The causes are suggested by the systematic bias in measuring the length of the nanowires. In the electronic measurement of the resistivity of a Mo nanowire (Zach *et al* 2000 *Science* **290** 2120–3), the proposed new method automatically identified the importance of accounting for the Ohmic contact resistance in the model of the Ohmic behavior in nanoelectronics experiments. The 95% confidence interval of resistivity in the proposed one-step procedure is determined to be $3.57 \pm 0.0274 \times 10^{-5}$ ohm cm, which should be a more reliable and precise estimate. The statistical quantification technique should find wide applications in obtaining better estimations from various systematic errors and biased effects that become more significant at the nanoscale.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Since the discovery of carbon nanotubes (CNTs) [3], people have realized that one-dimensional (1D) nanomaterials can exhibit dramatically different or enhanced properties from bulk materials. For example, CNTs are found to be the current strongest and stiffest materials in terms of tensile strength and elastic modulus respectively, both of which are about ten times larger than those of stainless steel [4, 5]. The 1D nanomaterials are believed to be the promising building blocks for micro-electro-mechanical systems (MEMS) and future nano-electro-mechanical systems (NEMS). In order to pave the path for further industrial applications, the need to characterize the physical and chemical properties of nanomaterials becomes urgent and important in current research.

However, accurately quantifying the mechanical, electrical and other properties of individual 1D nanomaterials is still a challenge to many existing testing and measuring techniques because of the following constraints. Firstly, the small size of the nanomaterials makes their manipulation rather

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difficult, prohibiting the application of well-established testing techniques for bulk materials. For instance, conventional tensile testing requires that the size of the sample be sufficiently large to be clamped rigidly by the sample holder without sliding. Thus, the conventional methods are not readily adapted to measure 1D nanomaterials. Secondly, the high purity and uniformity of nanomaterials has not been achieved yet. For example, single-walled carbon nanotubes can exhibit metallic or semiconductive properties depending on their different chiral vector [6]. Even nanomaterials with a simple structure such as ZnO nanowires/nanorods/nanobelts are expected to possess different properties with different growth orientations and dimensions [7, 8]. Thirdly, due to the actual small scale of nanomaterials, the experimental noise and bias become relatively large, making the measurement error significant and not negligible in current research [4, 9]. This can lead to large deviations in the experimental results from different research groups. The common deterministic approaches for quantification on bulk materials may not be suitable for nanomaterials. Therefore, new methods and methodologies need to be developed to quantify the properties of individual nanomaterials appropriately.

The new methodology should have the capacity to accommodate the aforementioned various experimental artifacts and uncertainties. Statistical techniques, which readily incorporate uncertainties of data, can be a powerful tool to contribute in a solution to these kinds of problems and to provide a more accurate estimation of the physical parameters. Previously, statistical methods are proposed to thoroughly exploit a color diagram for displaying a large amount of complex experimental results of CdSe nanowires, nanobelts, and nanosaws synthesis [10, 11]. We also made an early effort in the development of a new statistical modeling technique for quantifying the elastic deformation of a bridged ZnO nanobelt [12, 13]. These two specific cases, involving particular and complex situations, show the promising efforts of applying statistical techniques to improve the data analysis and quantification. To further broaden this vision systematically and arouse awareness in the nanoscience field, this research aims to use a few relatively simple but important examples to illustrate the capacity of employing statistical techniques in nanomechanics and nanoelectronics.

2. A general physical–statistical model

In general, a physical model is used to quantify the data to estimate the target physical parameter. Starting from the physical model, we propose a physical–statistical model to accommodate various systematic errors and bias effects. Suppose the physical model is

$$y = R(\beta)x, \tag{1}$$

where y is the dependent variable (response) and x is the independent variable (predictor). Here β represents the physical parameter to be quantified. Such a physical model is deterministic and may not be able to incorporate various uncertainties. To deal with experimental data with errors and other noise factors, we propose a physical–statistical model as

$$y = y_0 + (R(\beta) + R_0)x + \varepsilon, \tag{2}$$

where ε is the random error. Here y_0 is the bias adjustment parameter and R_0 can be considered as a slope adjustment parameter [14]. The adjustment parameters can provide the flexibility to model various experimental artifacts and biases. The physical–statistical model is then used to fit the entire measurement results from different samples or different trials. Hence we can obtain one single value for estimating β, which is statistically more reasonable and precise. Moreover, the model is likely to provide some insightful information about the experiments or measurements. The use of the proposed physical–statistical model is illustrated through the following examples.

3. Nanomechanics example

The resonance method represents one of the most important and widely used techniques in nanomechanics. The scheme of these kinds of experiments is to excite a single nanostructure such as a carbon nanotube, ZnO nanowire, or Si nanowire, which is fixed at one end and free at the other end, into its resonance status by an external oscillating electric field with a tunable frequency in a transmission electron microscope (TEM), a scanning electron microscope (SEM) or air [1, 15, 16].

When the frequency of the external oscillating electric field matches the intrinsic resonance frequency of the nanowire, a maximum vibration magnitude of the nanowire can be observed (see figure 1(a)). A series of resonance frequencies with different harmonics will occur, and normally the first harmonic is easy to detect. The physical model in this resonance method predicts the resonance frequency of a nanowire follows:

$$f = \frac{\beta_1^2}{2\pi L^2}\sqrt{\frac{EI}{m}}, \tag{3}$$

where f is the resonance frequency, β_1 is a constant ($\beta_1 = 1.875$ for first harmonic), L is the length, E is the elastic modulus, I is the moment of inertia of cross section, and m is the unit length mass. Zhou *et al* (2006) tabled all the measurement and calculation results for individual ZnO nanowires, and provided a mathematical average $E = 108$ GPa [15]. A similar data treatment with tables or diagrams displaying all the measurement results with large deviations is common in nanoscience field [2, 7]. Taking potential artifacts and experimental biases into account, statistical techniques can be used to better analyze the data and explain the experiment more appropriately, thus they can provide more insightful information. The observed frequency may not match the physical model well, and the statistical model should take the initial bias into consideration. A simplified form of the proposed physical–statistical model (1) is proposed to explain the data:

$$f = f_0 + \left(\frac{\beta_1^2}{2\pi L^2}\sqrt{\frac{I}{m}}\right)\sqrt{E} + \varepsilon, \tag{4}$$

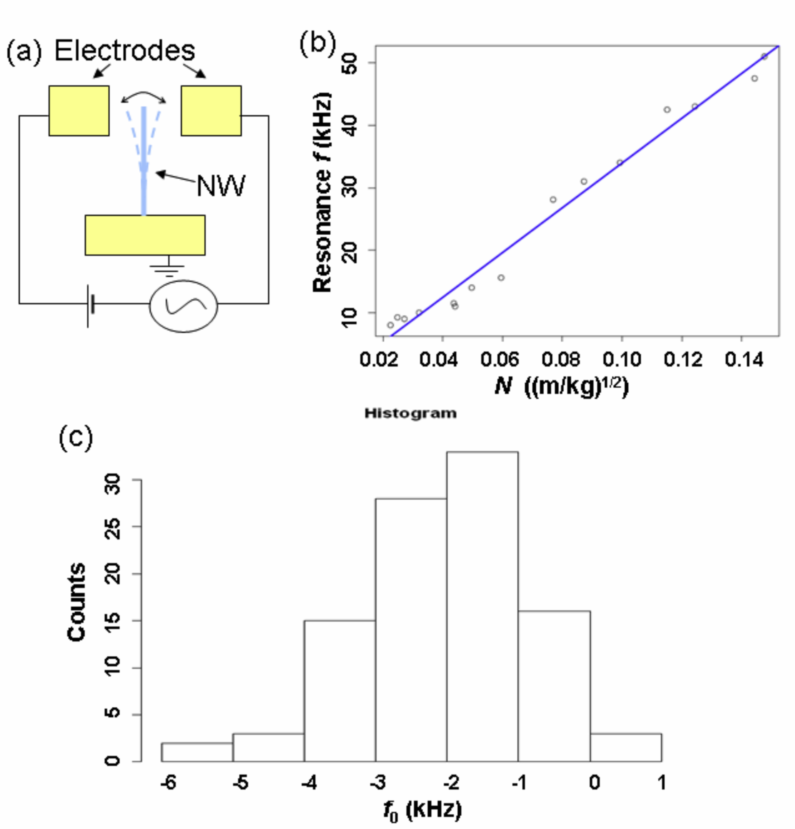

Figure 1. (a) The schematic diagram of the experimental setup for the resonance method. (b) The relationship between resonance frequency and new variable $N = (\frac{\beta_1^2}{2\pi L^2}\sqrt{\frac{I}{m}})$; the elastic modulus E can be inferred from the slope and the intercept indicates the negative f_0. (c) The histogram of the estimated f_0, calculated by randomly selecting half of the data points to perform a linear regression from model (4); this procedure is repeated 100 times.

where f_0 is an initial bias, ε is a random error, and the entire term inside the bracket is regarded as a new independent variable N. All discrete data are plotted in figure 1(b) to illustrate the relationship between the resonance frequency f and the new independent variable N. Assuming all the nanowires have the same elastic modulus, the slope of the linear fit of previous discrete data implies one physical parameter $\sqrt{E}$. From figure 1(b), we can obtain E = 128.33 GPa, $f_0 = -1.87$ kHz, and the standard error of the estimate E is 9.81 GPa. The value of E estimated by this statistical model is 18.8% bigger than the original mathematical average. The mechanical measurement results on ZnO nanowires still show some discrepancy, mainly because of the difference in the dimension, orientation, measurement and data treatment techniques. By accounting for the bias effects and random errors, the proposed method is to offer a more reliable estimate of the elastic modulus than the deterministic physical model.

f_0 is the bias adjustment parameter in (4) for modeling the resonance frequency f. It can provide some insights about the experiments and measurements. The negative value of f_0 may indicate that there are certain systematical biases occurred during the experiment. In practice, the systematical bias could come from various sources. Inconsistency in the cross section will statistically even out and become unimportant, and only the biased measured parameters will lead to biased results. One of these parameters is the length, which easily has a slightly smaller estimation in resonance methods because of the 3-dimensional to 2-dimensional projection effects. To the best of our knowledge, the projection effect on the final results obtained by resonance methods has not been discussed before which, however, is implied from the analysis using the proposed statistical techniques. The measured smaller value in the length results in a negative f_0, and a simple estimation of such a relation is provided in appendix A.1. Thus, it is very important to accurately measure the length, which was rather challenging for a free standing nanowire. Recently, an effective statistics-guided approach has been developed to better determine the lengths of randomly oriented nanowires under a microscope [17]. Our facile physical–statistical modeling strategy developed in this work can be more efficient and effective by adopting this approach of measuring the length.

Due to the limited number of data points, f_0 may not really exist and it could purely result from the statistical analysis. To further verify the existence of bias in the measuring length L resulting in the negative estimate of f_0, we conduct the following data analysis. We randomly select half of the data points to perform a linear regression from model (4) and get the estimate of f_0. The analysis is repeated 100 times and the

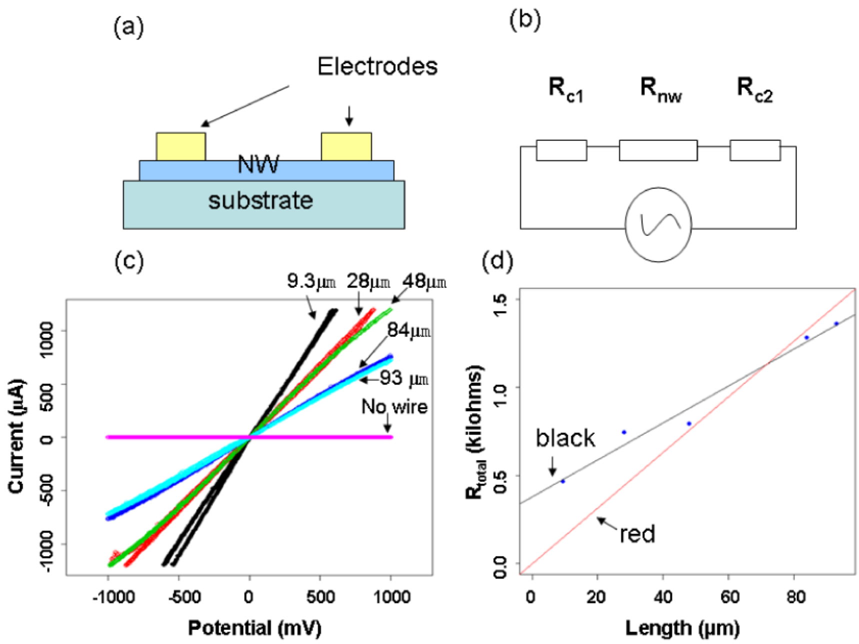

Figure 2. (a) The schematic diagram of the experimental setup for the electric measurement on a nanowire. (b) The simplified electrical diagram to represent (a). (c) The re-plot of the data for a 380 nm diameter Mo nanowire with a different length obtained from the work of Zach *et al* [2], black–9.3 μm, red–28 μm, green–48 μm, blue–84 μm, cyan–93 μm, magenta–no wire. (d) The correlation between the total resistance and the length of the nanowire. The red line is the fitting curve using model (5) and the black line is using model (6).

histogram of the estimated f_0 is shown in figure 1(c). From this figure, we can see that most of the estimated f_0 have negative values. This indicates that the observed length L tends to be smaller than the actual length, causing the elastic modulus E to be underestimated and unreliable using the physical model (3).

In addition, we can further theoretically show that even if the bias term y_0 does not exist and the model $y = \beta x + \varepsilon$ is valid, the linear regression method can achieve a more precise (smaller variance) estimate of β than that from the mathematical average. The technical derivation, which is a special case of Gauss–Markov theorem [18], is in the appendix A.2.

In this example, we have discussed the use of the discrete data points from different nanowires to estimate the mechanical parameter elastic modulus. In the next example, the proposed physical–statistical modeling and global fitting is used for all available quasi-continuous data curves such as the force–distance and current–voltage curves to estimate the electrical parameter resistivity. From the statistical point of view, this new approach is much more accurate and efficient.

4. Nanoelectronics example

In this example, nanoelectronics refers to the electric phenomena occurring in nanomaterials such as carbon nanotubes, ZnO nanobelts and Si nanowires. Figures 2(a) and (b) indicate the common configuration of a 1D nanomaterial electronic device. Although most 1D nanomaterial electronic devices might generally follow the traditional physical models, certain aspects still need to be considered carefully. For example, people normally ignore the Ohmic contact resistance for bulk materials; however, the Ohmic contact resistance may be comparable to the resistance of the nanomaterials as the dimension shrinks and it should not be ignored without scientific verification. Furthermore, the nanomaterials, being more sensitive and vulnerable to environmental mechanical vibrations or electrical interferences, can display stronger noise behavior in their electric transport measurement. Such concerns require an elaborate and systematic data quantification technique, where statistical modeling and analysis can play an important role in this aspect. For an Ohmic behavior measurement, the simplest physical model for a uniform nanowire is

$$V = \rho \frac{L}{A} I, \tag{5}$$

where V is the voltage, ρ is the resistivity, L is the length of the nanowire, A is the area, and I is the current. Zach *et al* calculated the resistivity of nanowires by electrically isolating various lengths of a Mo nanowire [2]. They used a linear fit to obtain the resistance from each I–V curve, then the resistivity of the nanowires was estimated from those calculated resistances as a function of the nanowire length. For convenience, we denote this method as a two-step procedure. To account for the resistance of the nanomaterials in the model, we consider the following model

$$V = \left(\rho \frac{L}{A} + R_0\right) I + \varepsilon, \tag{6}$$

where R_0 is the contact resistance during the measurement and there should be no bias in voltage V because of the nature of electricity. ε is a random error for the measurement data. Figure 2(d) shows a linear relation between the R_{total} of the system ($R_{total} = R_{nw} + R_0 = R_{nw} + R_{c1} + R_{c2}$) and the length of the nanowire. Following the two-step procedure using (6), we can also estimate the resistivity of the nanowires

Table 1. The standard multivariable linear regression analysis for the electrical measurements on a Mo nanowire.

	Estimate	Std. error	t value	p value
V_0 (mV)	−0.114	0.642	−0.177	0.86
ρ (ohm cm)	3.57×10^{-5}	0.014×10^{-5}	257.05	$<2 \times 10^{-16}$
R_0 (kilo hms)	0.405	0.002	197.94	$<2 \times 10^{-16}$

from the slope shown in figure 2(d), assuming R_0 is a constant. (Note: from table 1, later we can see that R_0 really has a small deviation.) We denote such a method as a refined two-step procedure. Clearly it shows that the fitting using (6) (black line, with the slope estimate 3.67×10^{-5} ohm · cm) is much better than that using (5) (red line, with the slope estimate 5.69×10^{-5} ohm · cm). From the data analysis using the proposed refined two-step procedure, the contact resistance parameter R_0 is statistically significant with a p value of less than 2×10^{-16}. The p value in statistics is a criterion to show the evidence of rejecting null hypothesis (the estimate is zero). It indicates that the Ohmic contact resistance can be comparably significant for nanoscale experiments, and cannot be neglected from this data analysis of nanoelectronics.

As we mentioned, the proposed physical–statistical method can use all available discrete data or quasi-continuous curves to quantify the targeted physical parameter. Therefore, we are proposing a general physical–statistical model to quantify that data with a one-step treatment instead of the previous two-step procedure. Moreover, we can calculate the Ohmic contact resistance and determine its significance from the specific physical–statistical model,

$$V = V_0 + \left(\rho \frac{L}{A} + R_0\right) I + \varepsilon. \tag{7}$$

For the data consisting of the series I–V curves for the same Mo nanowire with different lengths (i.e., by electrically isolating various lengths of this nanowire), there are only three unknowns V_0, ρ and R_0 regarded as parameters in the model. Thus, the equation (7) can be rewritten as:

$$V = V_0 + \rho\left(\frac{LI}{A}\right) + R_0 I + \varepsilon, \tag{8}$$

where LI/A and I are two independent variables. The parameters can be estimated in one step using all the data points. The standard multivariable linear regression analysis can provide the estimation of V_0, ρ and R_0 [19], which are summarized in table 1. The t value in table 1 is the value of the t-statistic (= estimate/standard error) for the hypothesis testing of whether the estimate is statistically different from zero.

From the table, the value of the estimate V_0 is very small compared with the observed voltage values. It is also statistical insignificant, which confirms that there is no bias in V because of the nature of electricity. The analysis also confirms that both the parameters ρ and R_0 are significant in the model (8). The significance of R_0 confirmed that the Ohmic contact resistance cannot be easily be ignored in this nanoelectronics experiment.

Furthermore, we can compare the estimation efficiency of ρ between the refined two-step procedure from model (6) and the one-step procedure from model (7). The 95% confidence interval of ρ in the refined two-step procedure is $3.67 \pm 0.686 \times 10^{-5}$ ohm cm, whose interval length is much larger than the 95% confidence interval in the one-step procedure $3.57 \pm 0.0274 \times 10^{-5}$ ohm cm. We can see that the estimates of ρ from the two proposed methods are close, but the one-step method gives a more precise estimation with much smaller standard errors.

The previous example illustrates a data treatment on I–V curves with Ohmic behavior. On the contrary, nonlinear I–V curves that origin from the Schottky metal–semiconductor junctions or PN junctions are also very common in nanoelectronics. For example, Lao *et al*'s work on ZnO nanobelts indicated a Schottky diode behavior [20]. Thus, the current should follow a diode current formula:

$$I = I_0\left[\exp\left(e\frac{V - V_{th}}{nkT}\right) - 1\right], \tag{9}$$

where the parameter n is the ideality factor of interest. In this situation, a log transformation can easily make a physical formula into a linear equation $\log(I)(I = \log(I_0) + e(V - V_{th})/nkT - 1$. The proposed method can then be used to apply on this linear model and then we can use repeated I–V curves to identify the ideality factor of the devices. In cases where the physical model is complicated and cannot be easily transformed into a linear formula, we can consider the possibility of the nonlinear regression models, which may not be in the scope of this work. Useful references can be found in some statistics literatures [21].

5. Conclusions

This work proposes a physical–statistical modeling approach and a global fitting statistical method to utilize all available discrete data or quasi-continuous curves to quantify a few target physical parameters. It can accommodate various experimental uncertainties and use all available information to provide more accurate, efficient and reasonable characterization. A few examples illustrate the applications of the proposed statistical quantification techniques in nanomechanics and nanoelectronics. In the resonance method for measuring the elastic modulus of ZnO nanowires, a negative f_0 is identified by our quantification technique and it might result from the systematic bias in measuring the length of nanowires. In the electronic measurement of resistivity of a Mo nanowire, the proposed new method automatically identified the importance of accounting for the Ohmic contact resistance in the model of the Ohmic behavior in nanoelectronics experiments. It can therefore give a more reliable and precise estimate of the resistivity. The statistical quantification technique should find wide applications in obtaining better estimations from various systematic errors and biased effects that become more significant at the nanoscale.

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Appendix

A.1.

For simplicity, L_m denotes the measured length, L_r is the real length, and ΔL is the small difference between L_m and L_r. It is easy to deduce the following steps:

$$\begin{aligned} f &= \frac{\beta^2}{2\pi L_r^2}\sqrt{\frac{EI}{m}} = \frac{\beta^2}{2\pi (L_m+\Delta L)^2}\sqrt{\frac{EI}{m}} \\ &= \frac{\beta^2}{2\pi L_m^2}\left(1-\frac{2\Delta L}{L_m}\right)\sqrt{\frac{EI}{m}} \\ &= -\frac{\beta^2}{2\pi L_m^2}\sqrt{\frac{EI}{m}}\left(\frac{2\Delta L}{L_m}\right)+\frac{\beta^2}{2\pi L_m^2}\sqrt{\frac{EI}{m}} \end{aligned}$$

A 3% bias in the length measurement can lead to approximately 1.5 kHz bias in f_0 for a nanowire with a 24 kHz resonance frequency, possibly leading to the 18.8% difference in elastic modulus measurements.

A.2.

Suppose the underlying model is

$$Y = \beta X + \varepsilon, \qquad \varepsilon \sim N(0, \sigma^2),$$

where β is the parameter of interest. There are n observations $(x_1, y_1), \ldots, (x_n, y_n)$. If we have two methods to estimate the parameter β, one (denoted by $\hat\beta_1$) is $\hat\beta_1 = \frac{1}{n}\sum_{i=1}^n \frac{y_i}{x_i}$, and the other (denoted by $\hat\beta_2$) is $\hat\beta_2 = \sum_{i=1}^n x_i y_i / \sum_{i=1}^n x_i^2$. Note that $\hat\beta_1$ is a mathematical average estimate used in Zhou *et al*'s work and $\hat\beta_2$ is the least-square estimate from the regression method. Obviously, both estimates are unbiased. i.e.,

$$E(\hat\beta_1) = \frac{1}{n}\sum_{i=1}^n \frac{E(y_i)}{x_i} = \frac{1}{n}\sum_{i=1}^n \frac{\beta x_i}{x_i} = \beta,$$

$$E(\hat\beta_2) = \frac{\sum_{i=1}^n x_i E(y_i)}{\sum_{i=1}^n x_i^2} = \frac{\sum_{i=1}^n x_i^2 \beta}{\sum_{i=1}^n x_i^2} = \beta.$$

Next, we check the variance of the two estimates $\hat\beta_1$ and $\hat\beta_2$. It is easy to show that

$$\operatorname{var}(\hat\beta_1) = \frac{1}{n^2}\sum_{i=1}^n \frac{\operatorname{var}(y_i)}{x_i^2} = \frac{1}{n}\sum_{i=1}^n \frac{1}{x_i}\sigma^2,$$

$$\operatorname{var}(\hat\beta_2) = \frac{\sum_{i=1}^n x_i^2 \operatorname{var}(y_i)}{(\sum_{i=1}^n x_i^2)^2} = \frac{1}{\sum_{i=1}^n x_i^2}\sigma^2.$$

Using the Cauchy–Schwarz inequality, i.e.,

$$\left(\sum_{i=1}^n a_i b_i\right)^2 \leqslant \left(\sum_{i=1}^n a_i^2\right)\left(\sum_{i=1}^n b_i^2\right),$$

we can get,

$$n^2 = \left(\sum_{i=1}^n \frac{1}{x_i}x_i\right)^2 \leqslant \left(\sum_{i=1}^n \frac{1}{x_i^2}\right)\left(\sum_{i=1}^n x_i^2\right)$$

$$\Rightarrow \frac{1}{n^2}\left(\sum_{i=1}^n \frac{1}{x_i^2}\right) \geqslant \frac{1}{\sum_{i=1}^n x_i^2}.$$

That is to say $\operatorname{var}(\hat\beta_1) \geqslant \operatorname{var}(\hat\beta_2)$. Therefore, we show that the estimate $\hat\beta_2$ is better (smaller variance) than $\hat\beta_1$.

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