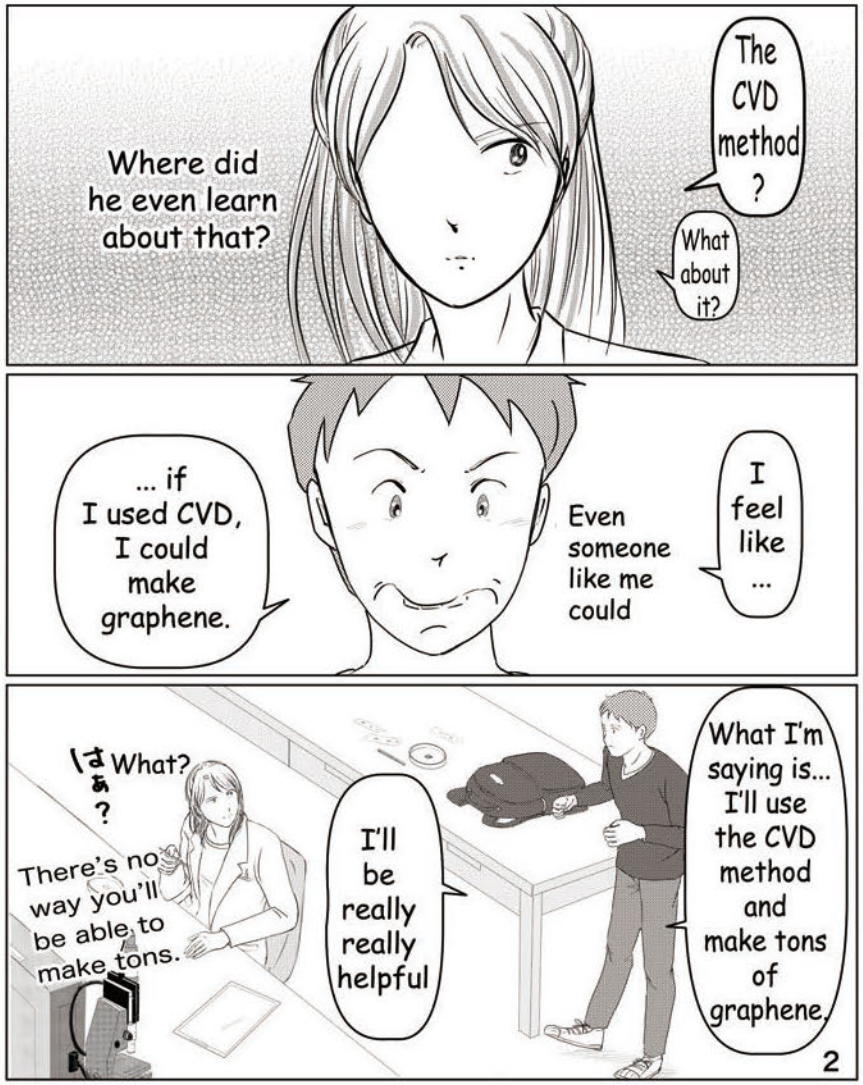
Chapter Two: CVD for Redemption



令和3(2021)年度学術変革領域研究(A)
2.5次元物質科学：
社会変革に向けた物質科学のパラダイムシフト
2.5D Materials
NEWS LETTER

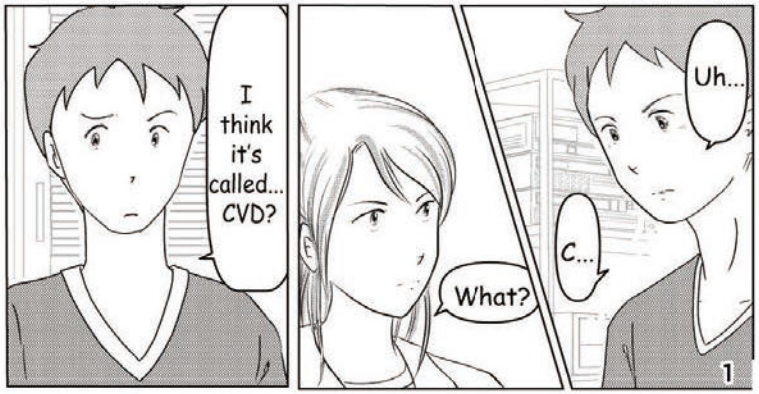
Welcome to the 2.5D Laboratory

Previously in the series

Koji Ota, freshly a senior at university, requests an assignment to the Ando Research Lab, drawn to the idea of being able to do cutting-edge science.

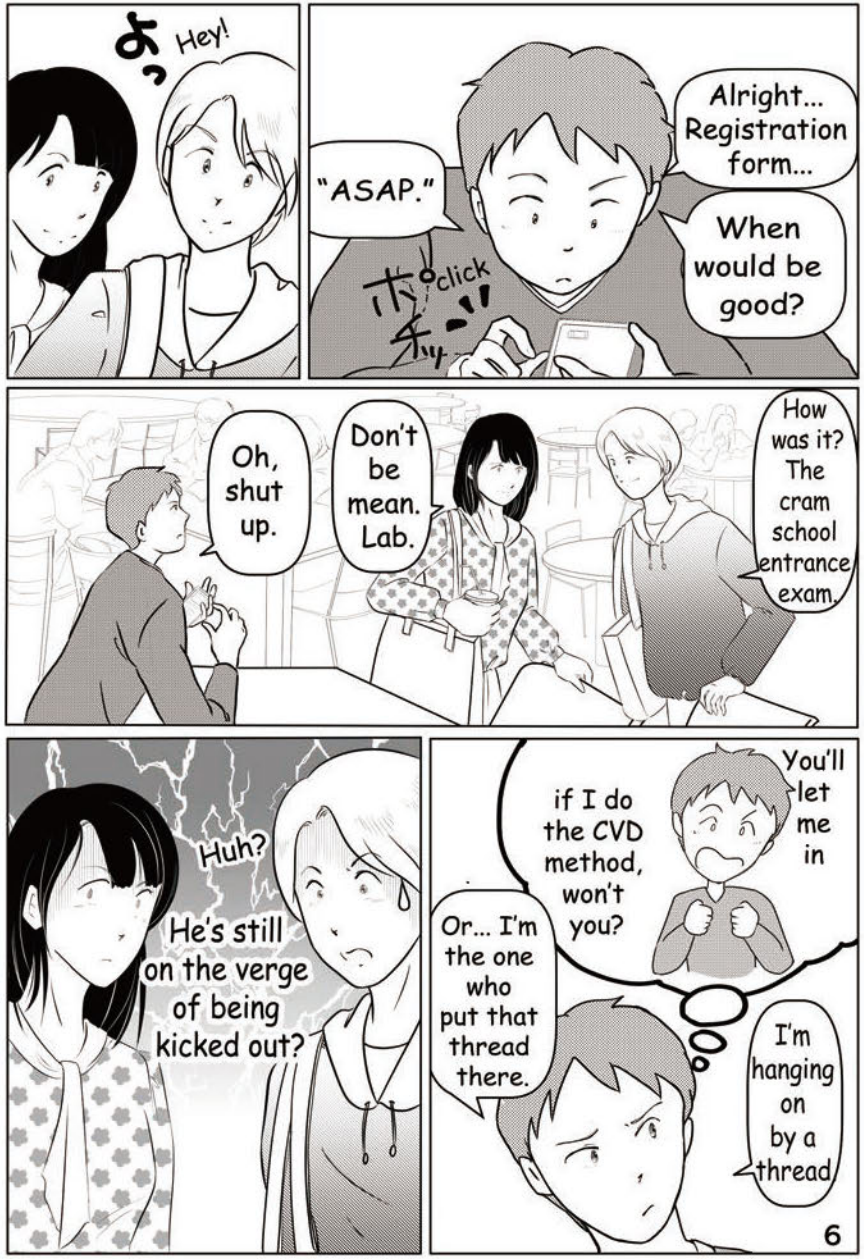
It turns out, however, that this is a deeply unpopular lab, with zero applicants over the past few years, and the only student a very strange graduate student.

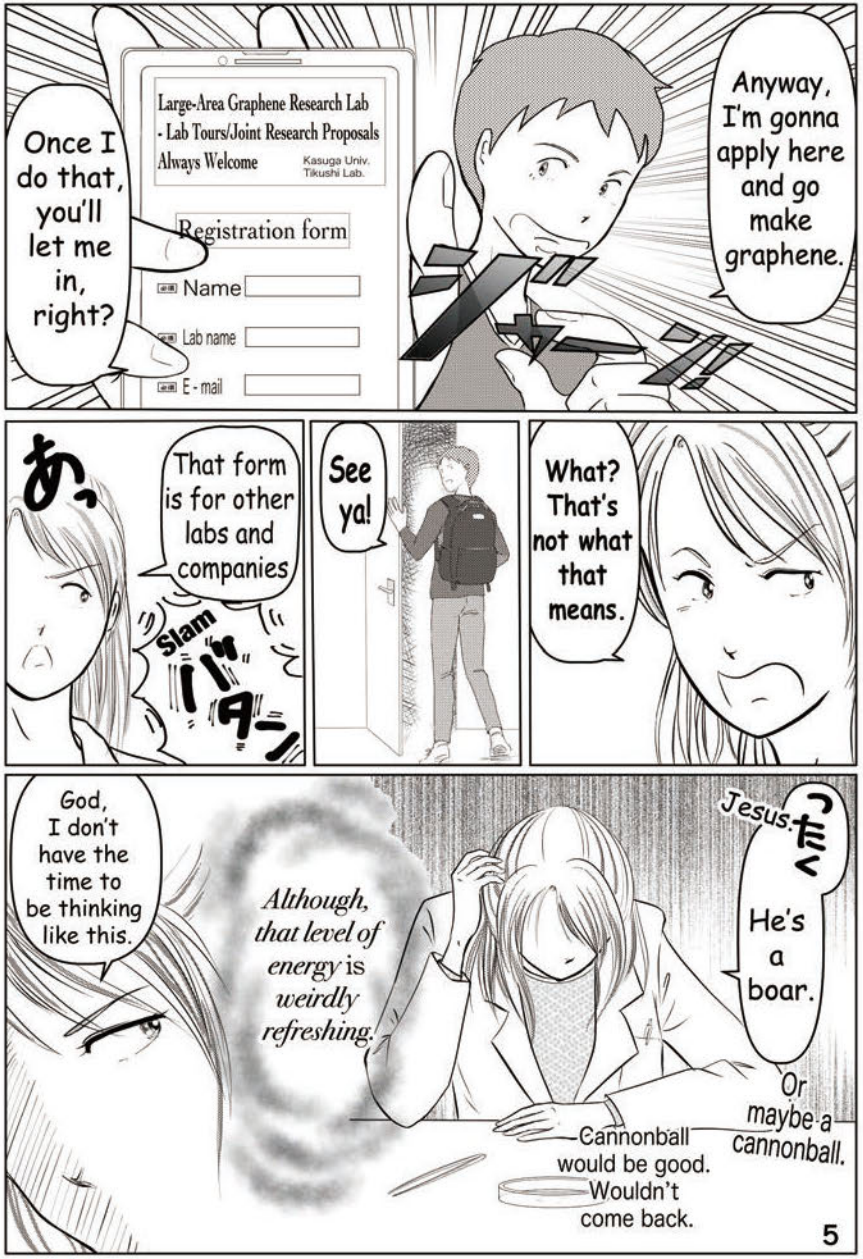
As if this weren't enough, Ota also fails to pass the entrance test for the lab—making graphene using the exfoliation method—and finds himself at a crossroads.

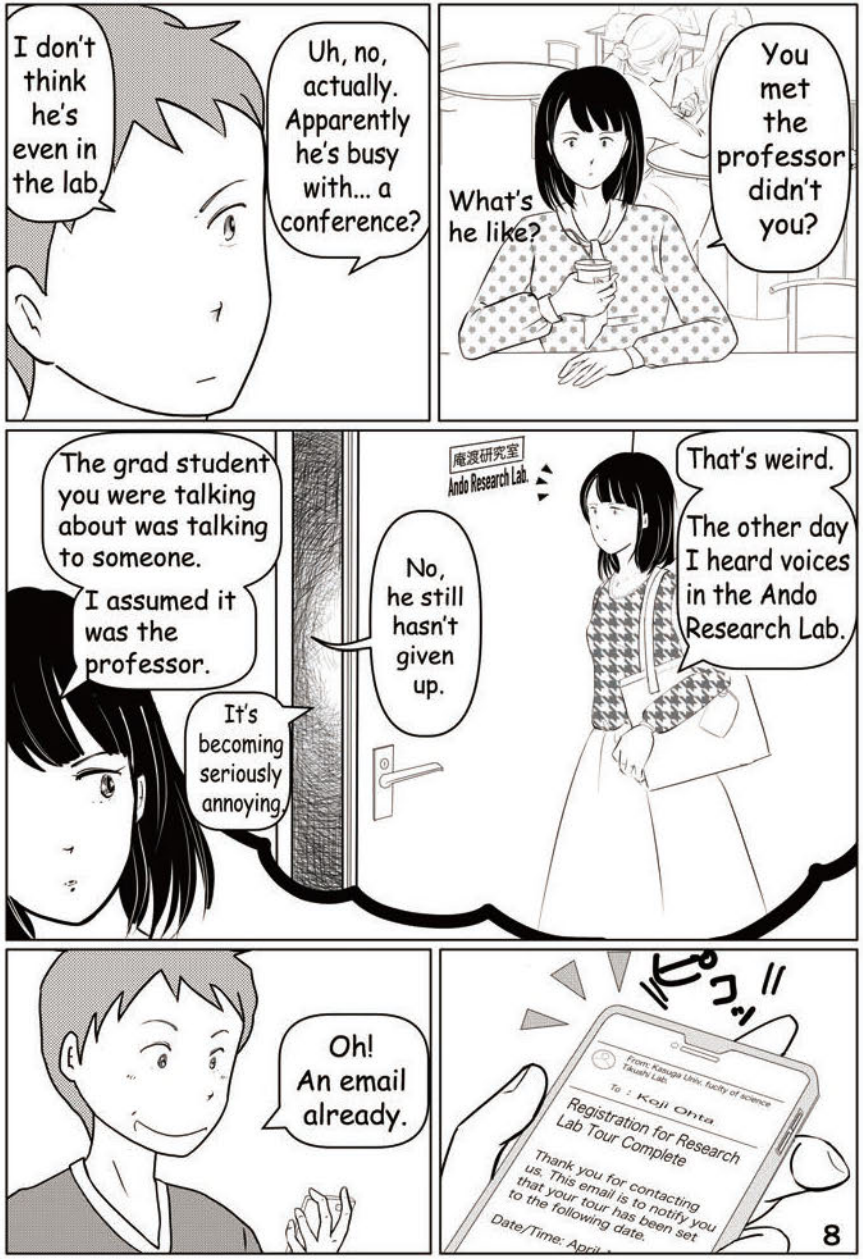



See ya!
We don't need
Please go write your thesis somewhere else.
ほら
Lool at this!
the kind of person who can't even make graphene using the exfoliation method.
Large-Area Graphene Set for Practical Application
Kasuga University Takes Step Forward by Advancing CVD Method
This university!
Tap Tap さっ さっ
Wait. But, but...
Excuse me? We don't need your advice.
There are other universities that are researching large-area graphene.
You might fall behind.
That's okay with you?
4


We don't need any clumsy people. And a total beginner at that.
You don't have to worry about us.
And we have all that we need.
おうち サイズ
House size
Car size
車サイズ
Sensor size
センサー サイズ
Smart-phone size
スマホ サイズ
To make it more practical for future use?
We gotta make it bigger... don't we?
Come on!
We're still just researching its electrical conductivity and photophysical properties, and all its other properties so small is fine.
I told you, we have enough
3


Hey!
Alright... Registration form...
"ASAP."
click
When would be good?
Oh, shut up.
Don't be mean. Lab.
How was it? The cram school entrance exam.
Huh?
He's still on the verge of being kicked out?
You'll let me in if I do the CVD method, won't you?
Or... I'm the one who put that thread there.
I'm hanging on by a thread
6

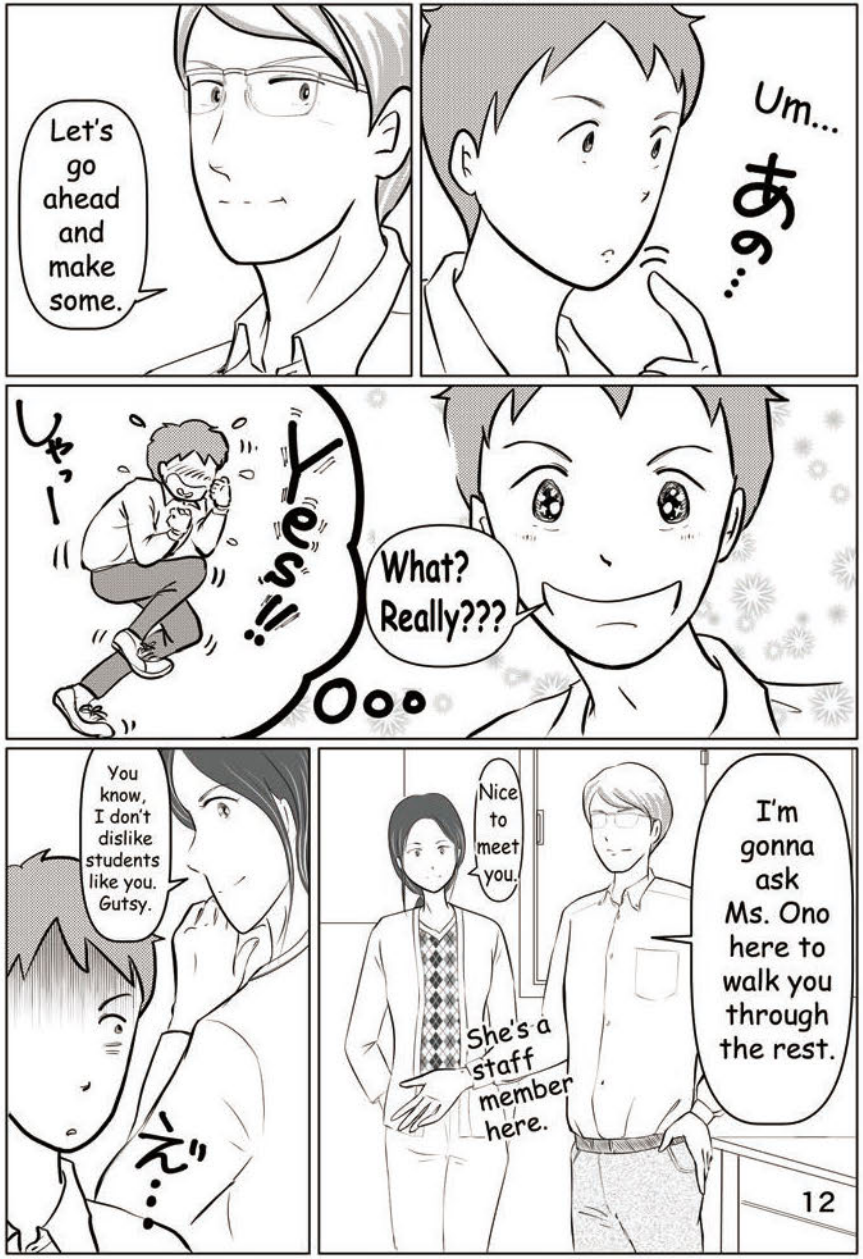

Large-Area Graphene Research Lab
- Lab Tours/Joint Research Proposals
Always Welcome
Kasuga Univ. Tikushi Lab.
Registration form
Name
Lab name
E-mail
Once I do that, you'll let me in, right?
Anyway, I'm gonna apply here and go make graphene.
That form is for other labs and companies
Slam
See ya!
What? That's not what that means.
God, I don't have the time to be thinking like this.
Although, that level of energy is weirdly refreshing.
Jesus.
He's a boar.
Or maybe a cannonball.
Cannonball would be good. Wouldn't come back.
5

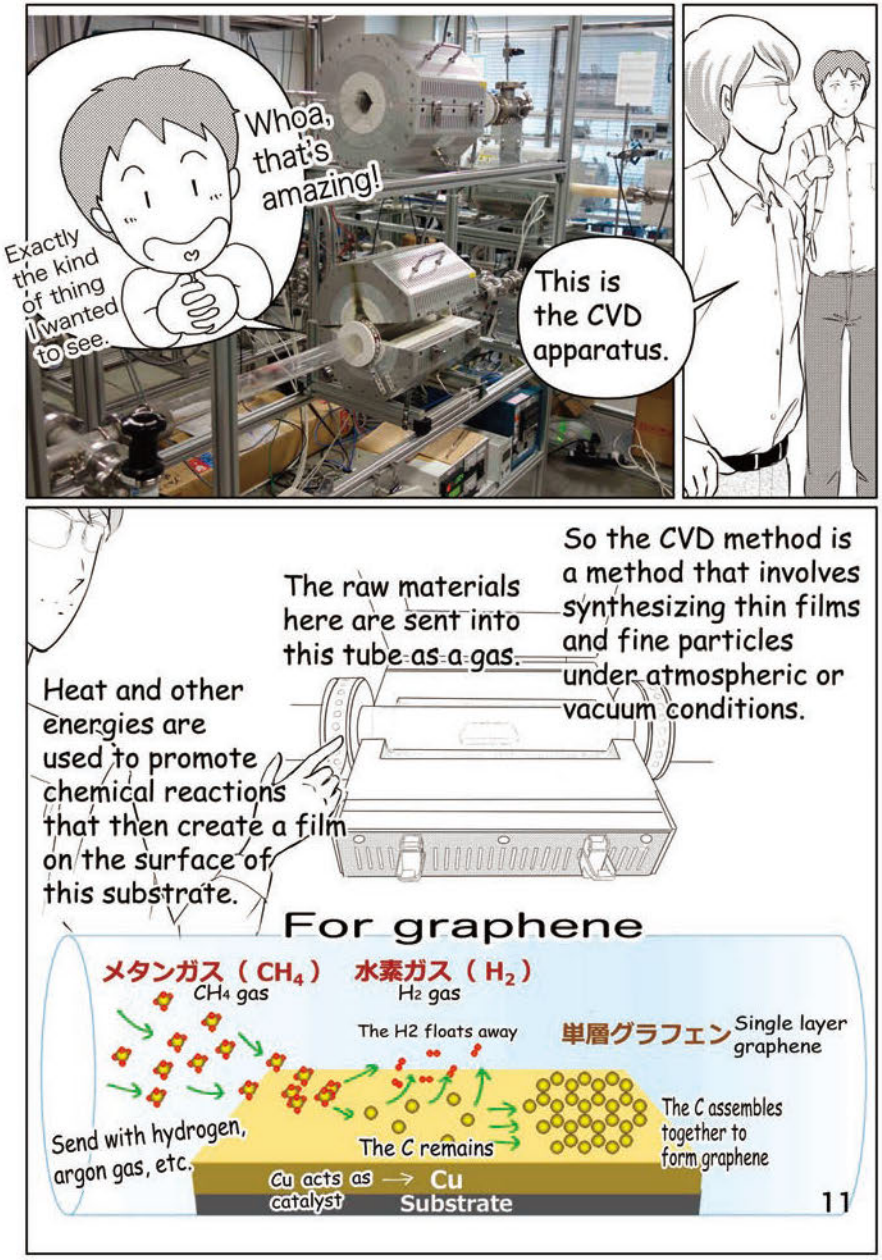

I don't think he's even in the lab.
Uh, no, actually. Apparently he's busy with... a conference?
What's he like?
You met the professor didn't you?
The grad student you were talking about was talking to someone.
I assumed it was the professor.
It's becoming seriously annoying.
庵渡研究室
Ando Research Lab.
No, he still hasn't given up.
That's weird.
The other day I heard voices in the Ando Research Lab.
Oh! An email already.
ピコッ
From: Katsuga Univ. Tuchi of science Takeshi Lab
To : Koji Ota
Registration for Research Lab Tour Complete
Thank you for contacting us. This email is to notify you that your tour has been set to the following date.
Date/Time: April
8


I can't make graphene if it means I have to look at it through a microscope.
Just listen! I. Am. Clumsy.
Hey! It got on me too!
Ah! うわ!!
Shaking your separatory funnel too hard and spraying the liquid everywhere...
Ups!
ガっ
Ended up having to do the experiment for the Meissner effect on a sample.
And during that superconductivity experiment, when you scattered the powder for the disk all over the place...
ゴロン
True. Your clumsiness is pretty legendary.
俺も被災したぞ I've been a victim to it.
So that's why your lab isn't decided yet...
Oh!
That's why I want to redeem myself using this CVD thing. Something where I'm less likely to fail.
7


Day of visit to Tikushi Research Lab, Kasuga University
Please, come in.
Hey, thanks for coming all this way.
Hi. I'm Koji. From Suruga University.
あ、Um, thank you for agreeing to this tour.
I've... never actually met him so I don't know.
Huh? You know Professor Ando?
How is Professor Ando? Is he doing well?
Of course.
new mail
accepted
sent
Request for Lab Tour
Dear Professor Tikushi,
It's been a long time.
A student of mine has decided all of a sudden to register for a tour of your lab. He is a graphene novice, so I ask that you please train him well.
Thank you.
Oh? Huh.
10


Man, you do have a lot of energy. Just energy.
Yeah
すご Whoa
You're gonna go to that lab?
No way. You just applied! That was so fast.
Was that last bit unnecessary?
I'm gonna learn how to make graphene and join that lab!
I am gonna be a graphene master!
グラフェンマスターになる俺はなる!!
Uh, what's happening?
Okay, okay.
You really don't know when to give up, do you?
9


Let's go ahead and make some.
Um...
あの…
Yes!!
What? Really???
You know, I don't dislike students like you. Gutsy.
えぇ…
Nice to meet you.
She's a staff member here.
I'm gonna ask Ms. Ono here to walk you through the rest.
12


Whoa, that's amazing!
Exactly the kind of thing I wanted to see.
This is the CVD apparatus.
So the CVD method is a method that involves synthesizing thin films and fine particles under atmospheric or vacuum conditions.
The raw materials here are sent into this tube as a gas.
Heat and other energies are used to promote chemical reactions that then create a film on the surface of this substrate.
For graphene
メタンガス（CH_4）
CH_4 gas
水素ガス（H_2）
H_2 gas
The H2 floats away
単層グラフェン
Single layer graphene
Send with hydrogen, argon gas, etc.
The C remains
The C assembles together to form graphene
Cu acts as catalyst
Cu Substrate
11

I mean, there's graphene out there that's in the range of 100 meters.

If we wanted to make it larger though, we could.

This one will be about 1.5 x 1.5 cm.

うっしゃ～ ゼミ残留 まちがいなし！

Yes!!! There's no way I'm not getting in now.

I see!

Of course they'll be bigger than when you use the exfoliation method.

ぐぬ～ Ah, my hands are shaking now.

Oh, and be careful not to scratch the substrate with the tweezers.

Set the substrate with the copper film on the mounting base.

Grabber?

Use this to set it in the tube.

うっしっし

No way we are letting you make nice crystals!

キレイな結晶なんて作らせないぞ～

And remember there's carbon everywhere. Don't let it contaminate the substrate.

Thank you.

Well, good luck.

This lets us make large, high-quality graphene.

In our lab, we use a sapphire substrate with an additional copper film deposited on top.

This is the copper film that will serve as the substrate.

Copper film deposited on top

上に銅を製膜しておく

Cu

CVD

単層グラフェン

This monolayer graphene has high crystallinity with uniform thickness, and its hexagonal lattice is highly oriented.

下はサファイアなどの単結晶基板

Single-crystal substrate such as sapphire on the bottom

Snap

ギュー

The size of this substrate?

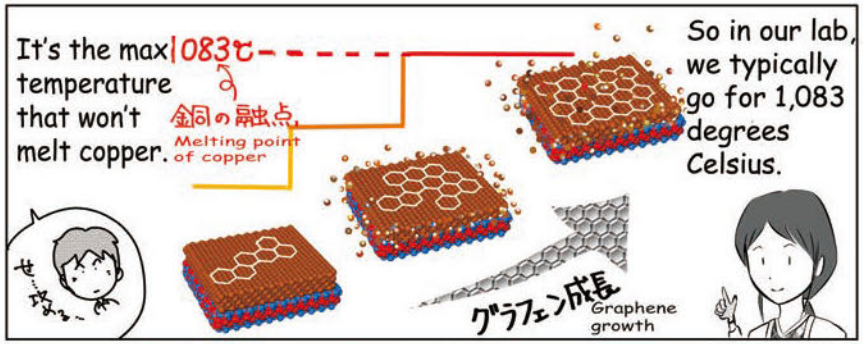
その基板の大きさってどのくらいになるんですか？

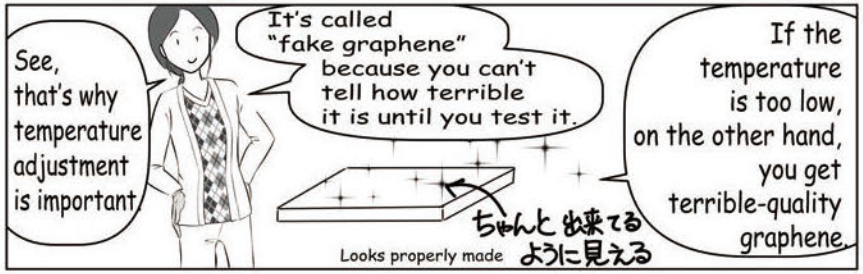
By large, how large do you mean?

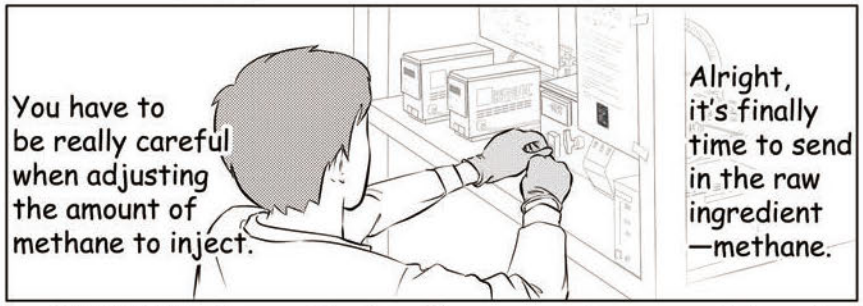
It's just that I've only ever made microscope-sized graphene, so I was wondering how big this one would be.

No! No. That's not it.

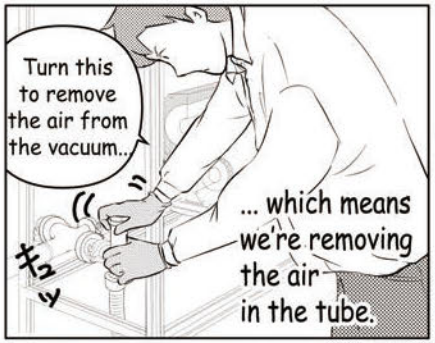
Oh, is that not enough for you?

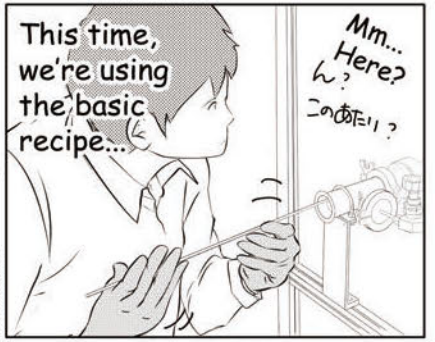

It's the max temperature that won't melt copper.
1083℃
銅の融点
Melting point of copper
So in our lab, we typically go for 1,083 degrees Celsius.
グラフェン成長
Graphene growth


See, that's why temperature adjustment is important.
It's called "fake graphene" because you can't tell how terrible it is until you test it.
If the temperature is too low, on the other hand, you get terrible-quality graphene.
ちゃんと出来てるように見える
Looks properly made

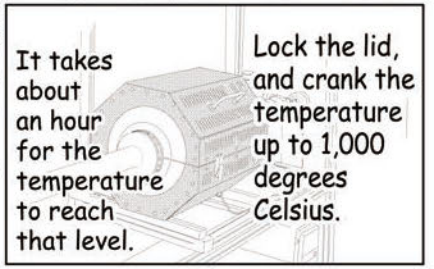

You have to be really careful when adjusting the amount of methane to inject.
Alright, it's finally time to send in the raw ingredient —methane.

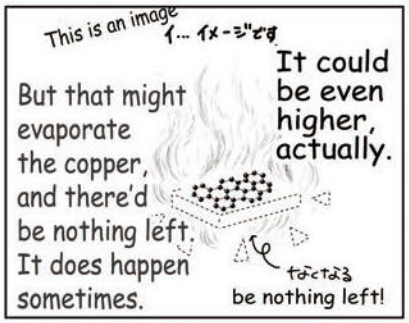

Oh no, there's more?
Get the amount wrong, and...

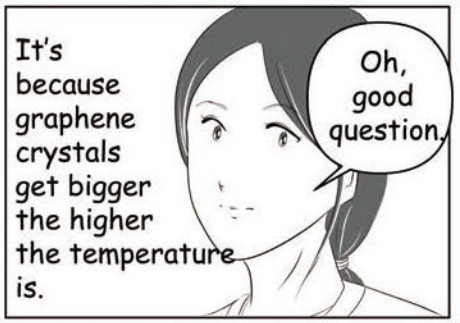

Turn this to remove the air from the vacuum...
キュッ
... which means we're removing the air in the tube.

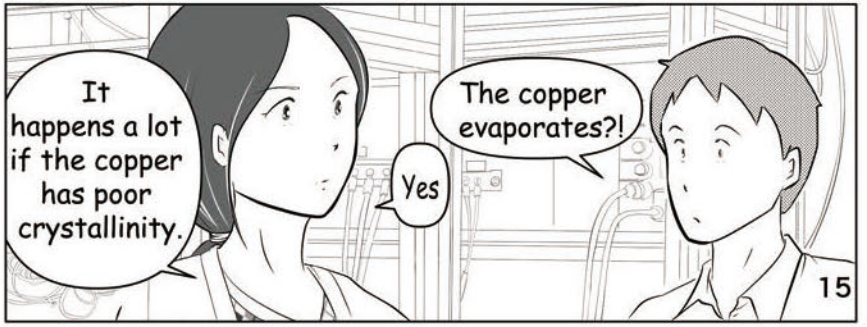

This time, we're using the basic recipe...
Mm... Here?
ん？ この辺に！？

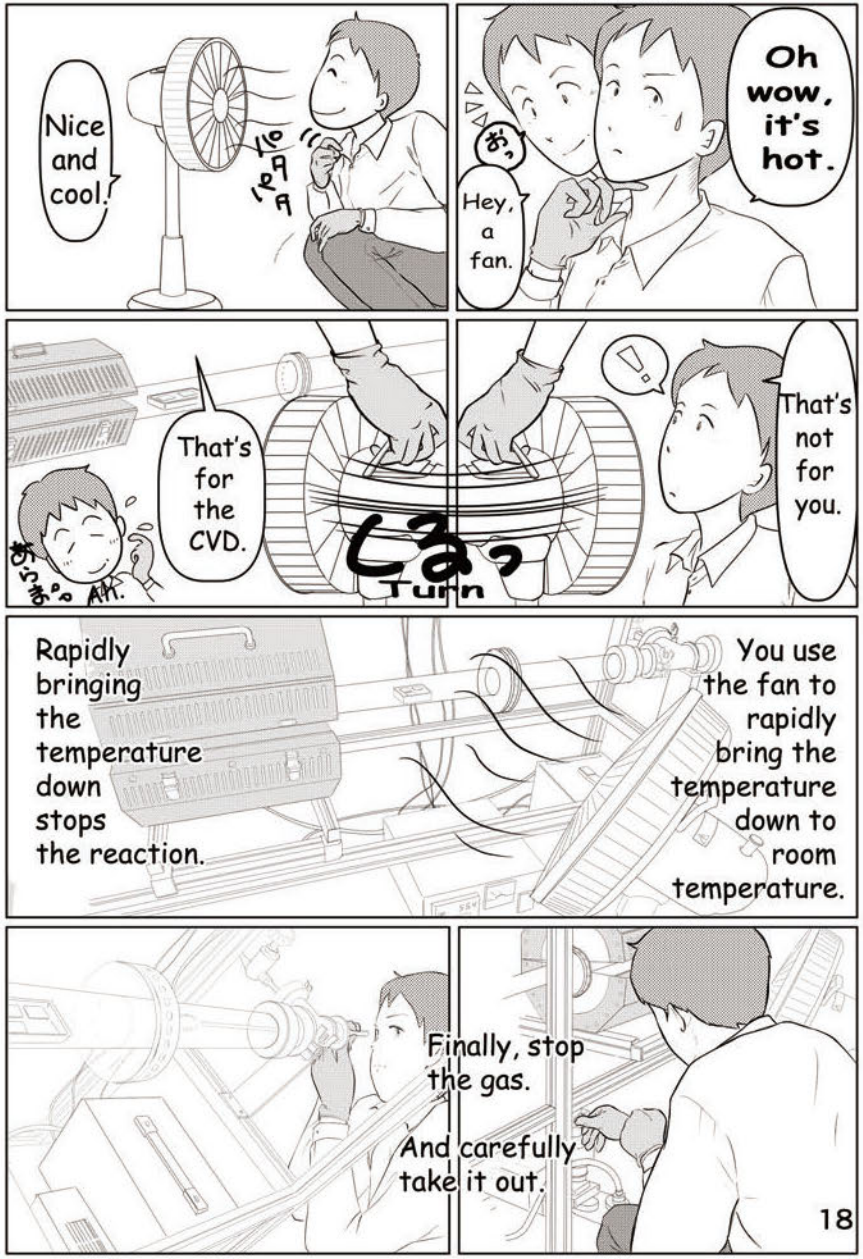

Why 1,000 degrees Celsius?


It takes about an hour for the temperature to reach that level.
Lock the lid, and crank the temperature up to 1,000 degrees Celsius.


This is an image
イ… イメージです
But that might evaporate the copper, and there'd be nothing left. It does happen sometimes.
It could be even higher, actually.
なくなる
be nothing left!

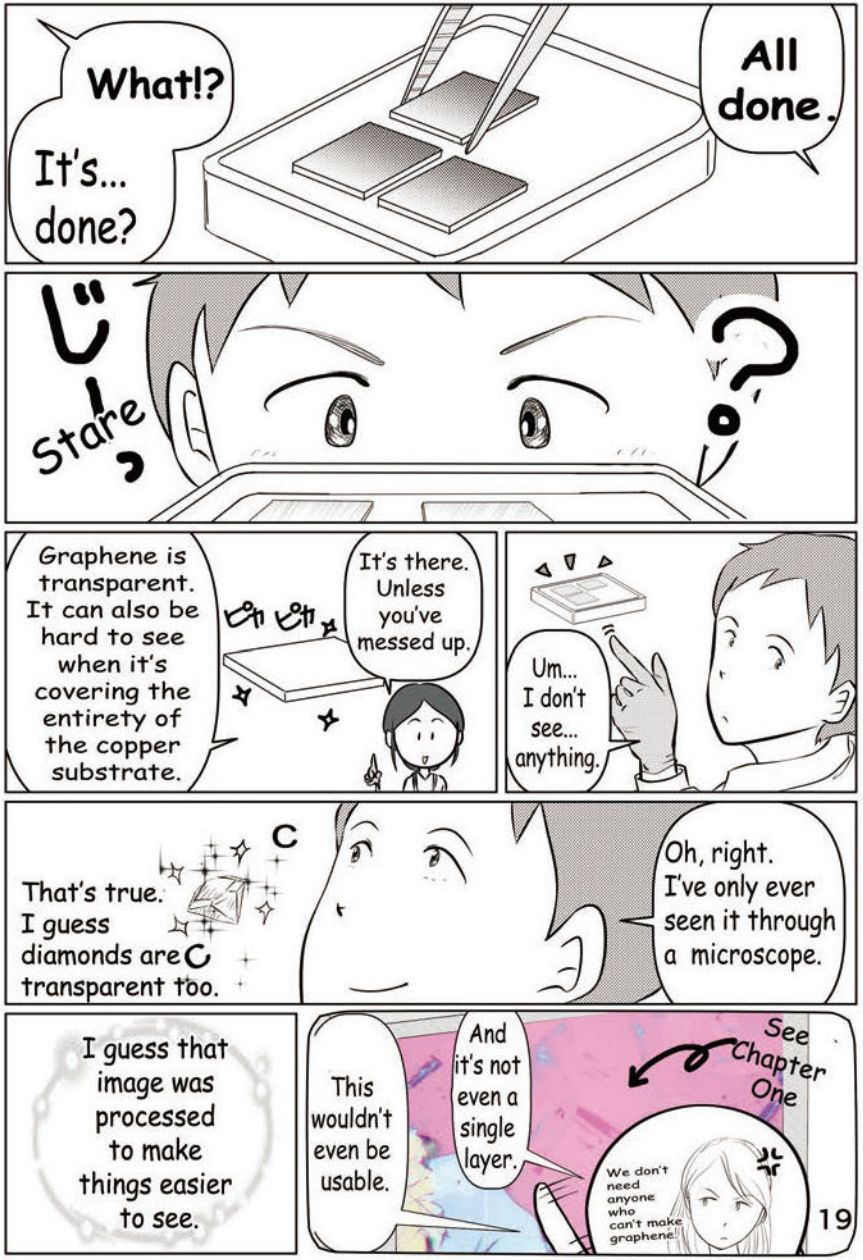

It's because graphene crystals get bigger the higher the temperature is.
Oh, good question.

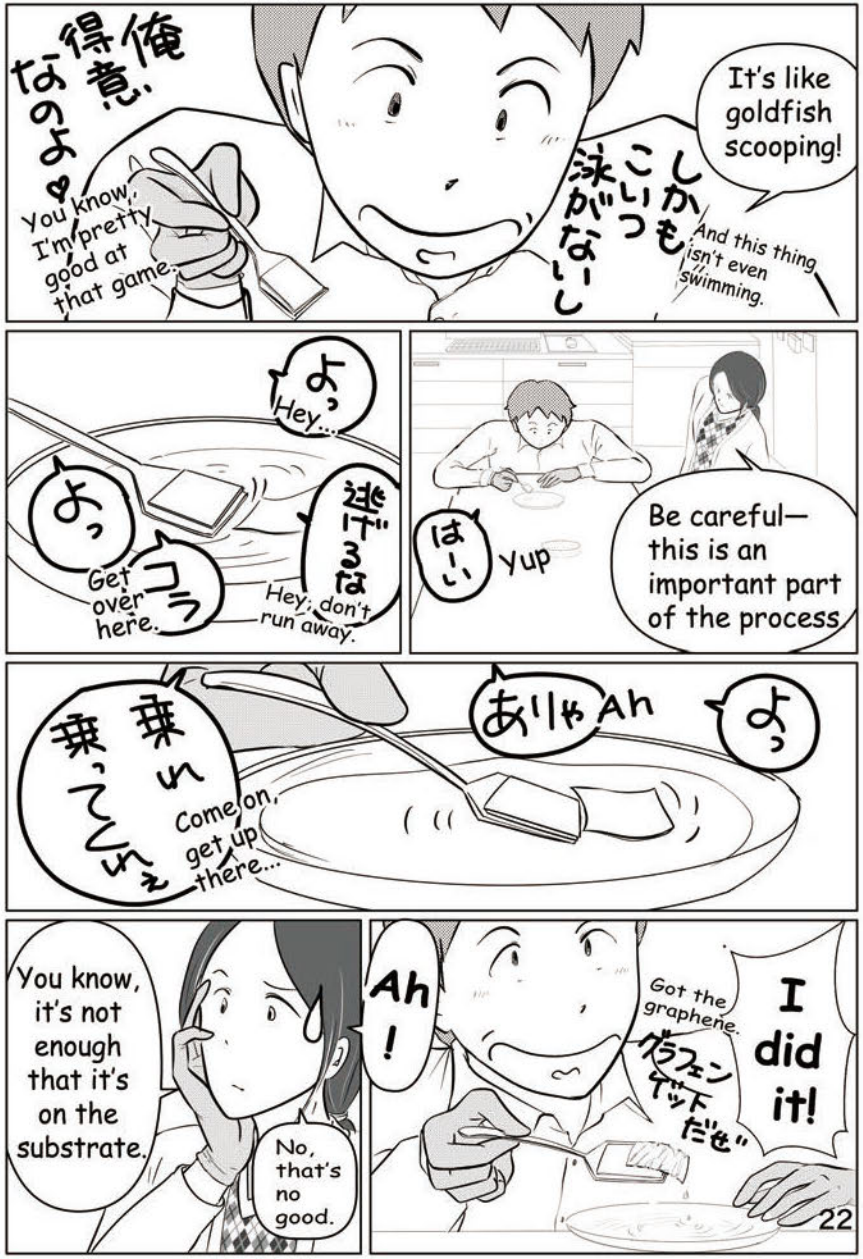

It happens a lot if the copper has poor crystallinity.
Yes
The copper evaporates?!

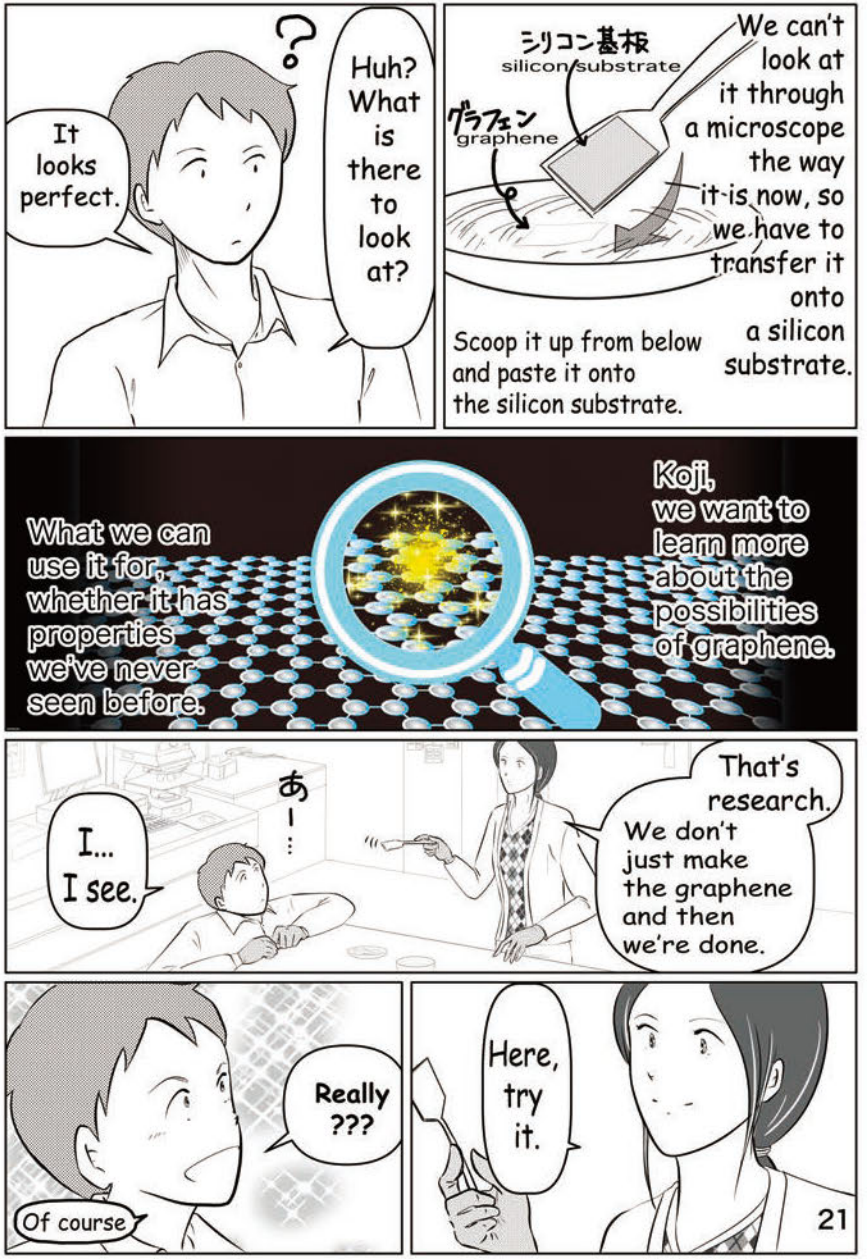

Nice and cool.
Oh wow, it's hot.
Hey, a fan.
That's for the CVD.
Ah.
Turn
That's not for you.
Rapidly bringing the temperature down stops the reaction.
You use the fan to rapidly bring the temperature down to room temperature.
Finally, stop the gas.
And carefully take it out.
18


Hahaha
Either the graphene fails to synthesize at all, or you just get a little piece.
There's the basic recipe, of course, but every lab has their own original way of doing things. That's part of the research.
It's these experiences and these little techniques you learn over time that are really important
open the lid, and slide the furnace over...
I think that's enough.
Stop the methane injection,
ホヤ ホヤ...
it's hot.
Freshly baked graphene...
おおお～
Oh wow
17

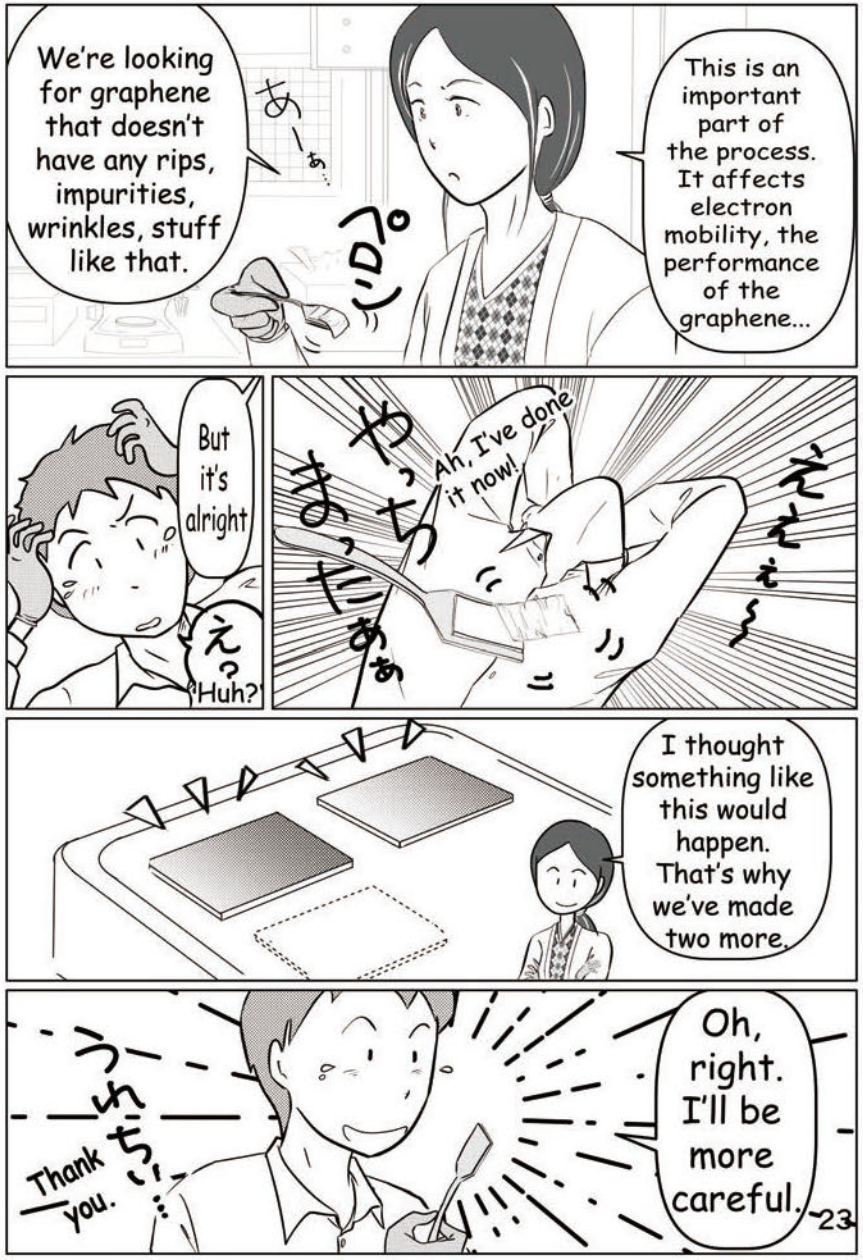

Whaaat? It's only an atom thick.
I can peel it off?
Alright. Next we'll be removing just the graphene.
Put it in this etchant※1 and the copper dissolves, leaving just the graphene.
Yup, you can.
※1 liquid used to chemically remove unwanted parts
※2 Actually takes several hours for copper foil
※2
Whoa, that's pretty. It's clear!
Scoop it up with this, and...
See that? It's floating up.
銅が溶けた液
Liquid with dissolved copper
It's so big! I don't need a microscope!
I-It's amazing.
Wash it with distilled water.
I'll be able to stay!


What!? It's... done?
All done.
じー Stare
Graphene is transparent. It can also be hard to see when it's covering the entirety of the copper substrate.
It's there. Unless you've messed up.
Um... I don't see... anything.
That's true. I guess diamonds are C transparent too.
Oh, right. I've only ever seen it through a microscope.
I guess that image was processed to make things easier to see.
This wouldn't even be usable.
And it's not even a single layer.
See Chapter One
We don't need anyone who can't make graphene!


俺得意なのよ♪
You know, I'm pretty good at that game.
しかもこいつ泳がないし
And this thing isn't even swimming.
It's like goldfish scooping!
よっ
Hey...
逃げるな
Hey, don't run away.
コラ
Get over here.
はーい
Yup
Be careful— this is an important part of the process
乗れ 乗ってくれぇ
Come on, get up there...
ありゃ Ah
You know, it's not enough that it's on the substrate.
No, that's no good.
Ah!
Got the graphene. グラフェンゲットだぜ
I did it!
22


It looks perfect.
Huh? What is there to look at?
シリコン基板
silicon substrate
グラフェン
graphene
We can't look at it through a microscope the way it is now, so we have to transfer it onto a silicon substrate.
Scoop it up from below and paste it onto the silicon substrate.
What we can use it for, whether it has properties we've never seen before.
Koji, we want to learn more about the possibilities of graphene.
I... I see.
あー...
That's research. We don't just make the graphene and then we're done.
Of course
Really ???
Here, try it.
21


コラ
逃げるな!
Come over here.
よっ
Hey!
スル
ペタッ
Stick
スル
Hey…
よっ
スルッ
Once more...
No!
It stuck to the side of the plate.
あ!
It went back.
戻った!
あー
Oh!
Tilt the plate a little so that it goes back in the water.
フワァ…
Man, it's so hard to see.
Transparent goldfish!
ほっ
Phew
プッ
Pu!
Is it always this hard?


We're looking for graphene that doesn't have any rips, impurities, wrinkles, stuff like that.
あーぁ…
プロ
This is an important part of the process. It affects electron mobility, the performance of the graphene...
But it's alright
え?
Huh?
やっとあった
Ah, I've done it now!
I thought something like this would happen. That's why we've made two more.
うれしい!
Thank you.
Oh, right. I'll be more careful.

Supervised ; Group A01 Hiroki Ago, Professor & Aika Uchida, Staff, Global Innovation Center, Kyushu University,
For more information https://25d-materials.jp